

**2000 Performance Report
General Chemistry and
Microbiology Section**

February 2001

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2000 Performance Report General Chemistry and Microbiology Section

Bernie Wright, Priscilla Wong, Susan Janhurst
Laboratory Services Branch
Ontario Ministry of the Environment

February 2001

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2000 PERFORMANCE REPORT
GENERAL CHEMISTRY AND MICROBIOLOGY SECTION

Bernie Wright, Priscilla Wong, Susan Janhurst

Laboratory Services Branch

Ontario Ministry of the Environment

February 2001

INTRODUCTION

The General Chemistry and Microbiology Section (GCMS) is part of the Ministry of the Environment Laboratory Services Branch. The section is comprised of two units, they are the Water Chemistry and Microbiology Unit. The Water Chemistry Unit identifies and provides quantitative analysis for major ions, nutrients, and physical properties in a variety of matrices. The Microbiology Unit identifies and enumerates indicator bacteria of water and waste waters.

This report provides a brief outline of the analytical quality control (QC) program associated with sample analysis and examines 2000 performance data for each test in the Water Chemistry and Microbiology Units. GCMS strives to maintain a high standard of analytical performance through its quality assurance program. QC is an integral part of this process.

A number of changes have taken place in the General Chemistry and Microbiology Section since the 1999 performance report was issued. The following describes those changes.

METHODS DISCONTINUED BY GCMS.

- Chloride (E3148)
- Chloride (E3372)
- Chlorine, Total Residual (E3309)
- Conductivity (E3177)
- Nitrogen, Nitrate (E3148)
- Nitrogen, Nitrate (E3372)
- Sulphate (E3148)
- Sulphate (E3372)

DORSET METHODS TRANSFERRED TO ENVIRONMENTAL MONITORING AND REPORTING BRANCH (not included in 2000 report).

- Alkalinity, Gran (E3042)
- Alkalinity, Total Fixed Endpoint (E3042)
- Calcium (E3249)
- Carbon, Dissolved Inorganic (E3028)
- Chloride (E3147)
- Color, True (E3025)
- Conductivity (E3024)
- Magnesium (E3249)
- Nitrogen, Ammonia Plus Ammonium (E3374)
- Nitrogen, Nitrate Plus Nitrite (E3374)
- pH (E3042)
- Phosphorous, Total (E3036)
- Potassium (E3249)
- Sodium (E3249)
- Sulphate (E3147)
- Sulphate (E3372)

METHODS ADDED (MICROBIOLOGY).

Escherichia coli (E3226)
Escherichia coli (E3371)
Escherichia coli (E3407)
Faecal streptococci (E3371)
Heterotrophic Plate Count (E3408)
Indicator Organisms (E3226)
Pseudomonas aeruginosa (E3371)
Total Coliform (E3226)
Total Coliform (E3371)
Total Coliform (E3407)

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1.0 PERFORMANCE REPORT FORMAT

The parameters are those analysed by the GCMS for 2000.

The performance report is organized alphabetically according to test name (eg. Dissolved Organic Carbon is filed under the heading "Carbon, Dissolved Organic") and second, by the method reference number. Detailed information concerning the format of each page is outlined below:

1.1 TEST DESCRIPTION

<u>TITLE:</u>	The name of the test parameter.
<u>IDENTIFICATION:</u>	
Laboratory	Location where the test is performed.
Method Reference No:	A number assigned by the Quality Management Unit to an analytical test method eg.(E3370).
Product Code:	LIMS code for analysis request.
Sample Type/Matrix:	The various sample types that can be routed to the method.
Method Introduced:	Date that the method was implemented at the laboratory.
Reporting Units:	Unit of measurement in which the results are reported.
Supervisor:	Name of supervisor/manger responsible for the method.

SAMPLING:

The type of container and preservative (if applicable) that is used and minimum volume of sample that is usually required. Any sample preparation that is normally performed in the field, is also indicated (1).

SAMPLE PREPARATION:

Sample preparation techniques which are usually performed at the laboratory before analysis.

ANALYTICAL PROCEDURE:

Brief summary of the analytical method used to determine the parameter.

INSTRUMENTATION:

Type of instrumentation used to perform the test. Automated continuous flow systems consist of a sampler, peristaltic pump, manifold for reagent addition, detection system and readout system. Microcomputers are used to control the operation of analytical equipment and/or data acquisition.

REPORTING:

W and T are low level data qualifiers (2). A value reported as $\leq W$ is interpreted as not present, the value accompanying the remark is the lowest reportable value of the method under routine operating conditions. A value (multiple of W) reported as $\leq WE$ is interpreted as above for $\leq W$ following non-routine dilution of the sample to allow analysis of the target substance. A value reported as $< T$ is interpreted as target substance identified, use caution in interpretation unless more sample data supports this result. A value reported as $< TE$ is interpreted as above for $< T$ following non routine dilution of the sample to allow analysis for the target substance.

To provide a consistent LSB approach to data reporting, GCMS calculates W from the standard deviation of duplicates (S_2), near zero, by rounding down to the nearest 1,2 or 5 digit (4). T is five times W. The latest calculations, valid at date of publication for W and T values of all active methods, are contained in this report (APPENDIX A).

Data is reported to a maximum of three significant figures. Low level results are reported in increments of "W".

CALIBRATION:

The number of standards used to calibrate the analytical system plus blanks if applicable.

CONTROLS:

The calibration, drift, recovery, and interference controls that are used when applicable to ensure that the system is operating properly.

MODIFICATIONS:

Modifications made to the test in 2000.

NOTES:

Explanatory notes which may aid the data user in interpreting results and information.

1.2 PERFORMANCE DATA SUMMARY

QUALITY CONTROL DATA FROM/TO:

The period of time over which data were collected.

ANALYTICAL RANGE AND REPORTING UNIT:

The full scale value for the analytical range is given in concentration units.

CALIBRATION CONTROL:

Calibration control includes a table outlining the number of data collected over the selected time period, expected concentrations of the control standards, the calculated mean concentration of these standards, mean bias (mean concentration minus the expected concentration), and standard deviations of each control standard. The between run standard deviation (S), the within run standard deviation (S_w), the ratio S/S_w , and the control limits for standards sums and differences are provided.

RECOVERIES (Where applicable):

The table outlines the number of data collected over the selected time period, expected concentrations of the recovery standards, the calculated mean concentration of these standards, and standard deviations of each recovery standard.

DUPLICATES:

The table outlines within run duplicate data collected over the selected time period. Data are sorted into a number of concentration spans. The standard deviation for duplicates is provided for each range. The coefficient of variation (%) is determined by dividing the mean standard deviation (S_2) for a particular concentration span by the mean concentration of duplicate results in that span and multiplying by 100.

OTHER CHECKS (Where applicable):

The table outlines the number of data collected over a selected time period, the calculated mean concentration of ie., blank, and standard deviation.

1.3 QUALITY CONTROL GRAPHICS

CALIBRATION CONTROL:

Calibration control standards sums and differences are plotted on a horizontal scale for the period of data collection (referred to on the graphs as "QUALITY CONTROL STANDARD A+B" for example). The vertical scale consists of the warning/control limits expressed on either side of the expected value. These limits were chosen from analytical performance data.

2.0 ANALYTICAL QUALITY CONTROL PROGRAM

Quality control is a continuous process that involves constant checks of sample processing procedures. This report summarizes the QC data collected during analytical processing to monitor performance of the analytical system.

Calibration Standards are verified for identity, purity and concentration accuracy by comparison against independent sources wherever possible. A series of calibration standards are analyzed covering the analytical range. Calibration curve characteristics (slope, intercept and curvature) are examined on a per run basis as indicated by the method.

Once a system has been calibrated, quality control begins. Depending on the analytical procedure, quality control may be used to evaluate: calibration, blank, recovery, sensitivity, potential interference, and sample repeatability.

Calibration and Blank

Calibration is controlled by a minimum of two quality control standards and a long term blank which are prepared and maintained independently of the calibration standards. The system is not calibrated with the quality control standards. The long term blank is Pure De-ionized Water (Pure-DW) used to prepare the quality control standards and has zero concentration of the target analyte. Control standards are prepared less frequently than calibration standards and errors in newly prepared calibration standards can be detected by this cross check. Newly prepared control standards are run in parallel with in-use control standards and must meet control requirements over three consecutive runs before the new standards are accepted for routine use.

The standard deviation of the control standards is used to estimate the between-run standard deviation (S) and is compared against the within-run standard deviation (S_w). If the ratio S/S_w exceeds 1.5 then poor control of systematic error can be inferred (3). Values for S and S_w are calculated as follows:

$$2S^2 = (S_A)^2 + (S_B)^2 \qquad 2S_w^2 = (S_{A-B})^2$$

Where

S_A = standard deviation (s.d.) of control standard A
 S_B = s.d. of control standard B
 S_{A-B} = s.d. of the difference between control standards A and B

NOTE: If a second range is employed for a test, more control standards are used because, in many systems, the between-run standard deviations are concentration dependent.

Detailed description of the quality control processes are outlined in several LSB documents (2)(4)(5) and (6).

Control/Warning Limits

The control standards data are assessed and compared against the control/warning limits established from previous data to determine whether the calibration process is in control. The limits are set up initially based on method performance(4), and are reviewed when method and/or performance data reviews are conducted to determine if modifications are required based on historical data calculations. Control limits are calculated for the sums and differences of control standards (A,B,C,D) by the equations:

$(A+B) \pm 4(S_{A+B})$ for the sum of A+B

$(B+C) \pm 4(S_{B+C})$ for the sum of B+C

$(C+D) \pm 4(S_{C+D})$ for the sum of C+D

$(A-B) \pm 3(S_{A-B})$ for the difference of A-B

$(B-C) \pm 3(S_{B-C})$ for the difference of B-C

$(C-D) \pm 3(S_{C-D})$ for the difference of C-D

Note: Warning Limits are calculated by the same formulae above (using ± 2 instead of 4 and 3 respectively).

If a control limit is exceeded, the analysis is stopped, corrective action taken and the control standards are re-analysed.

Recovery

Some methods require sample pre-treatment, such as digestion or extraction. A recovery check, suitable to that method, is required to estimate the efficiency of the pre-treatment. Recovery standards are usually prepared at 0%, 20% and 80% of full scale. The solutions are analysed in the same manner as routine samples. Although these solutions are not used to calibrate the instrument, corrections for the blank and matrix effects are calculated and applied if necessary. For an analytical run to be accepted, the recoveries should be within $\pm(5\% + T/2)$ of their expected values. (See Section 1.1 "Reporting" for T determination). The average blank should be within three standard deviations of its historical mean. If a second range is employed for a test, at least one additional recovery standard is used.

Sensitivity and Baseline

Any change in the sensitivity of the instrumentation is monitored periodically during the run, as defined by the method, by analysing a standard that is usually 80% of full scale, and comparing the reading to the original calibration standards. Baseline drift is usually recorded by periodic analysis, as defined by the method, of Pure-DW which does not contain any of the analyte, but may be treated to correspond to sample pre-treatment.

Interference

The interference check is run on any test where a substance may be present in concentrations that affect the results. The check is carried out near the threshold concentration of the interfering substance, beyond which the methodological safeguards used to minimize the interference are no longer effective. The check indicates that the interference has no effect up to the specified concentrations.

Sample Repeatability

Generally, one sample out of twenty is analysed in duplicate up to a maximum of three duplicates per analytical run. The samples are selected for non-adjacent, within-run duplicate analyses. By analysing samples in duplicate, the ability of the analyst to obtain repeatable analytical results, within an analytical run, can be determined. For results to be acceptable, at least two of the three duplicate pairs must conform to limits that are set based on historical performance.

The observed differences in duplicate results are accumulated and sorted into 3 to 4 ranges based on analyte concentration. A standard deviation is calculated for each concentration range. The algorithm differs from the conventional standard deviation as follows:

Conventional Std. Dev. (1)*

$$S_1 = \sqrt{\frac{\sum_{i=1}^n (\bar{x} - x_i)^2}{n-1}}$$

Std. Dev. of Duplicates (2)*

$$S_2 = \sqrt{\frac{\sum_{i=1}^{n'} (x_1 - x_2)_i^2}{2n'}}$$

* Standard deviations used for the data summaries.

Where

S_1 = sample standard deviation

S_2 = duplicate difference standard deviation

n = number of data

$\bar{x}$ = mean of data

x_i = i^{th} result

$(x_1 - x_2)_i$ = difference of the i^{th} duplicate

n' = number of duplicate pairs

The standard deviation (S_2) of the duplicate difference is also expressed as the coefficient of variation (CV).

$$CV = \frac{S_2}{\bar{X}} \times 100$$

2.1 PERFORMANCE SUMMARIES

ACIDITY, GRAN

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/08/82
Method Reference No	E3248	Reporting Units	$\mu\text{eq/L as H}^+$
LIMS Product Code	PHACD3248	Supervisor	P. Wilson
Sample Type/Matrix	Effluent, Industrial Waste, Raw Sewage, Drinking Water, Leachate, Precipitation, Surface Water		

SAMPLING:

Quantity Required	50 mL
Container	Glass or Plastic

ANALYTICAL PROCEDURE:

Sample aliquots (50.0 mL) are titrated with 0.01 N sodium hydroxide to pH >8.3. The titrant is standardized against 0.0005 N potassium hydrogen phthalate. The titrant delivery rate is determined from the slope of the titration curve and the stability of the pH readings following each aliquot of titrant. Data are subjected to Gran analysis. pH and total fixed endpoint acidity are determined simultaneously.

INSTRUMENTATION:

Automated modular titration system with microcomputer control and data processing software.

REPORTING:

Maximum Significant Figures: 3	Current W value: 5	Current T value: 25
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CALIBRATION:

2 standard buffers covering the pH range of 4 to 9

CONTROLS:

Calibration	Long Term Blank plus 2 standards, e.g. QCA
-------------	--

NOTES:

Oct '99. The W value of 1 and T value of 5 were changed to 5 and 25 respectively based on historical data.

ACIDITY, GRAN (E3248)

QUALITY CONTROL DATA FROM 11/01/00 TO 25/10/00

Analytical Range: to 1000 µeq/L as H⁺

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	15	500.0	517.9	17.9	9.9990
B:	15	200.0	201.9	1.9	8.3825
A+B:		700.0	719.8	19.7	15.2150
A-B:		300.0	316.0	16.0	10.4403

s.d.(AB)

S(between runs): 9.23

Sw(within run): 7.38

S/Sw: 1.2

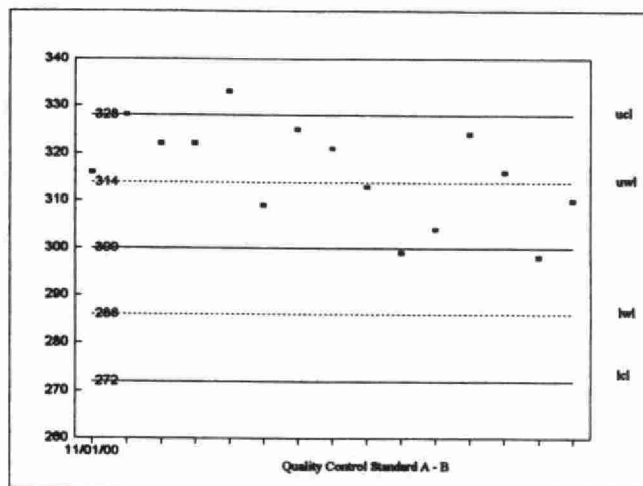
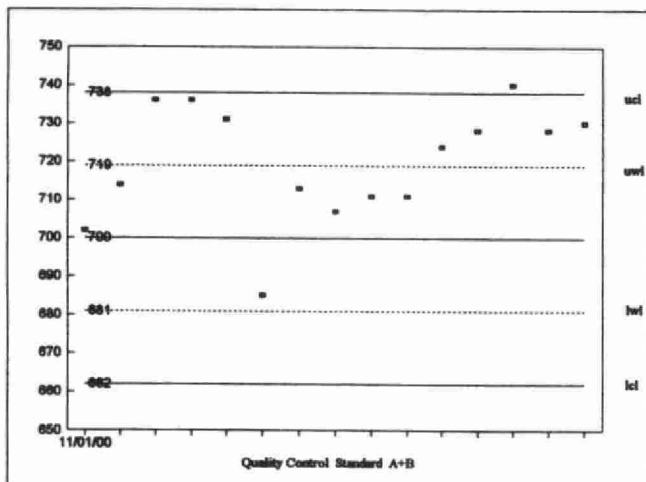
On any given day the calibration is accepted if the calibration control values obtained lie within the ranges:

662 - 738 for A+B
272 - 328 for A-B

DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
5	0 - 100	8.5264	37.2
16	101 - 250	3.9330	6.2
10	250 - 500	11.3886	7.1.
31	Overall	7.8453	

ACIDITY, GRAN ($\mu\text{eq/L}$ as H^+)
QUALITY CONTROL DATA FROM 11/01/00 TO 25/10/00
E3248



ACIDITY, TOTAL FIXED ENDPOINT

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/08/82
Method Reference No.	E3248	Reporting Units	mg/L as CaCO ₃
LIMS Product Code	PHACD3248	Supervisor	P. Wilson
Sample Type/Matrix	Effluent, Industrial Waste, Raw Sewage, Drinking Water, Leachate, Precipitation, Surface Water		

SAMPLING:

Quantity Required	50 mL
Container	Glass or Plastic

ANALYTICAL PROCEDURE:

Sample aliquots (50.0 mL) are titrated with 0.01 N sodium hydroxide to pH >8.3. The titrant is standardized against 0.0005 N potassium hydrogen phthalate. The titrant delivery rate is determined from the slope of the titration curve and the stability of the pH readings following each aliquot of titrant. pH and gran acidity are determined simultaneously.

INSTRUMENTATION:

Automated modular titration system with microcomputer control and data processing software.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.2	Current T value: 1.0
--------------------------------	----------------------	----------------------

CALIBRATION:

2 standard buffers covering the pH range of 4 to 9

CONTROLS:

Calibration	LTBL plus 2 standards, e.g. QCA
-------------	---------------------------------

NOTES:

Oct '99. The W value of 0.05 and T value of 0.25 were changed to 0.2 and 1.0 respectively based on historical data.

ACIDITY, TOTAL FIXED ENDPOINT (E3248)

QUALITY CONTROL DATA FROM 11/01/00 TO 25/10/00

Analytical Range: to 100 mg/L as CaCO₃

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	15	25.0	25.862	0.862	0.5898
B:	15	10.0	9.937	-0.063	0.4413
A+B:		35.0	34.659	-0.341	0.7345
A-B:		15.0	15.193	0.193	0.4620

s.d.(AB) S(between runs): 0.30

Sw(within run): 0.20

S/Sw: 1.5

On any given day the calibration is accepted if the calibration control values obtained lie within the ranges:

32.8 - 37.2 for A+B
13.4 - 16.6 for A-B

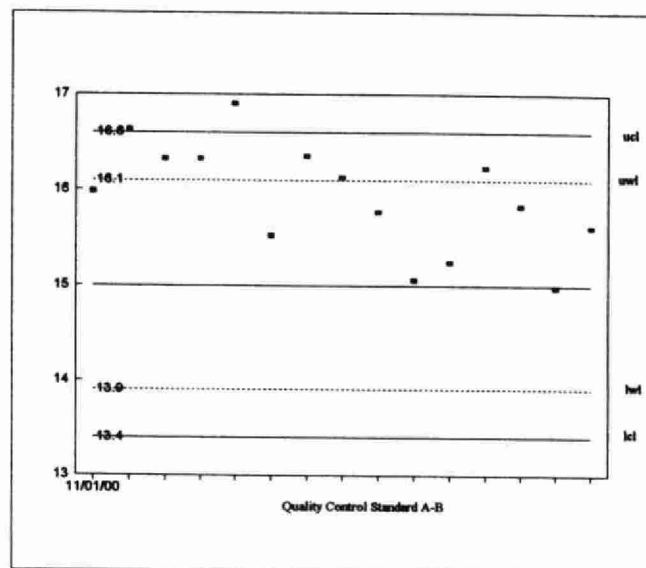
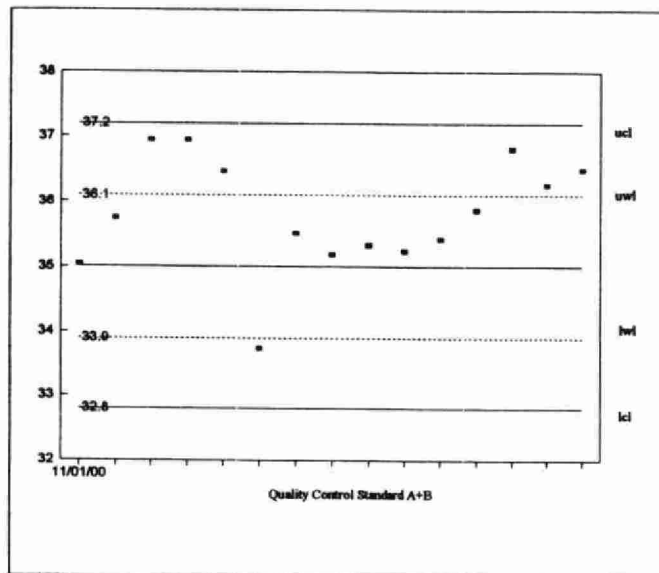
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
16	0 - 4.0	0.2430	8.6
8	4.1 - 10.0	0.0678	12.6
4	10.1 - 20.0	0.6223	5.2
28	Overall	0.4696	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	15	0.014	0.0327

ACIDITY, TOTAL FIXED ENDPOINT (mg/L as CaCO₃)
QUALITY CONTROL DATA FROM 11/01/00 TO 25/10/00
E3248



ALKALINITY, TOTAL FIXED ENDPOINT

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	09/07/80
Method Reference No.	E3218	Reporting Unit	mg/L as CaCO ₃
LIMS Product Code	PHALCO3218	Supervisor	P. Wilson
Sample Type/Matrix	Sludge, Effluent, Industrial Waste, Raw Sewage, Drinking Water, Ground Water, Leachate, Precipitation, Surface Water		

SAMPLING:

Quantity Required	50 mL
Container	Glass or Plastic

ANALYTICAL PROCEDURE:

Samples (10.0 mL) are titrated with 0.02 N sulphuric acid to pH endpoint of 4.5. The titrant delivery rate is determined from the slope of the titration curve and the stability of the pH reading following each aliquot of titrant.

pH, and conductivity are determined simultaneously.

INSTRUMENTATION:

Automated modular titration system with microcomputer control and data processing software.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.5	Current T value: 2.5
--------------------------------	----------------------	----------------------

CALIBRATION:

2 standard buffers covering the pH range of 4 to 9

CONTROLS:

Calibration	BL plus 4 standards, e.g. QCA
Drift	In run standards throughout the run (tap water diluted to 50% V/V)

NOTES:

May '97 the W value was changed from 0.2 to 0.5 after a review of 2 years low level duplicate data '94-95.

ALKALINITY, TOTAL FIXED ENDPOINT (E3218)

QUALITY CONTROL DATA FROM 05/01/00 TO 19/12/00

Analytical Range: to 1000 mg/L as CaCO₃

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	107	250	248.60	-1.40	2.3680
B:	107	100	99.70	-0.30	0.9634
C:	107	25	25.03	0.03	0.5489
D:	107	2.5	2.45	-0.05	0.1236
A+B:		350	348.31	-1.69	2.7514
A-B:		150	148.90	-1.10	2.3454
B+C:		125	124.74	-0.26	1.3182
B-C:		75	74.70	-0.33	0.8492
C+D:		27.5	27.48	-0.02	0.5384
C-D:		22.5	22.59	0.09	0.5858

s.d.(AB) S(between runs): 1.81

s.d.(BC) S(between runs): 0.78

s.d.(CD) S(between runs): 0.40

Sw(within run): 1.66

Sw(within run): 0.60

Sw(within run): 0.41

S/Sw: 1.1

S/Sw: 1.3

S/Sw: 1.0

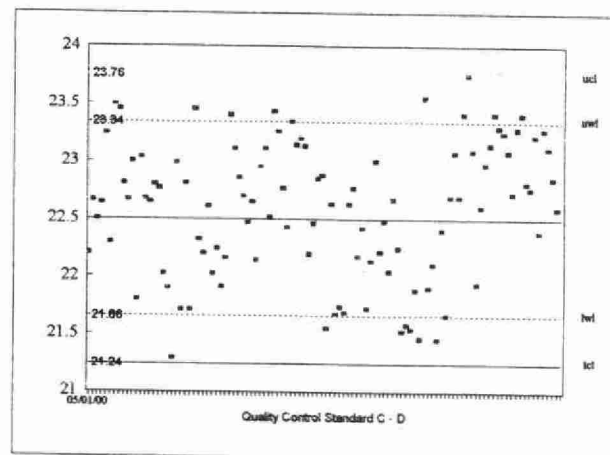
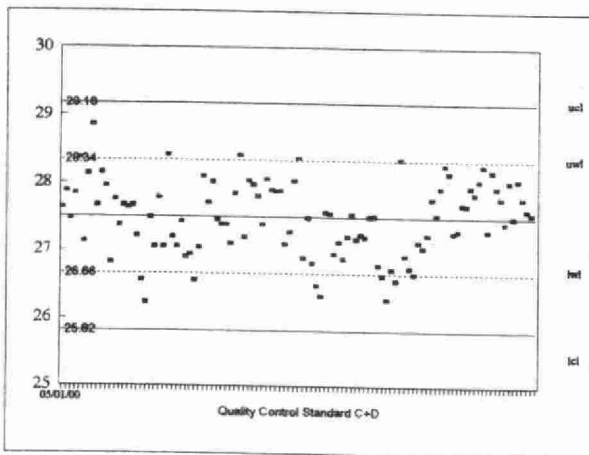
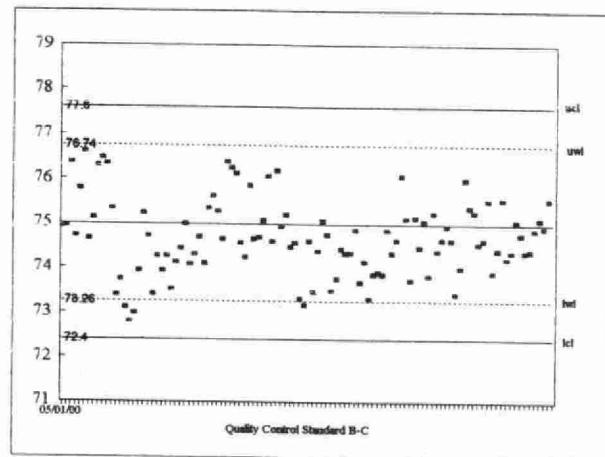
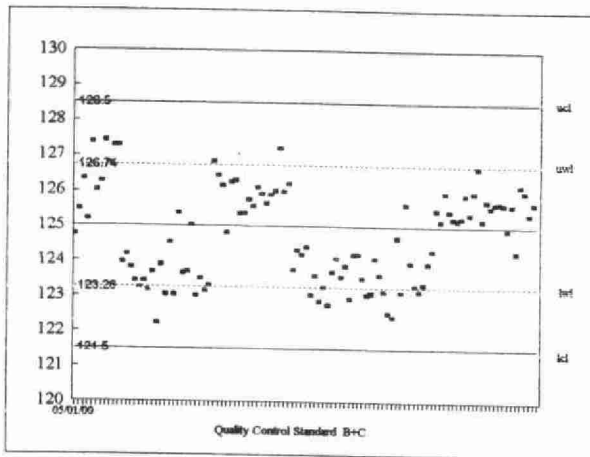
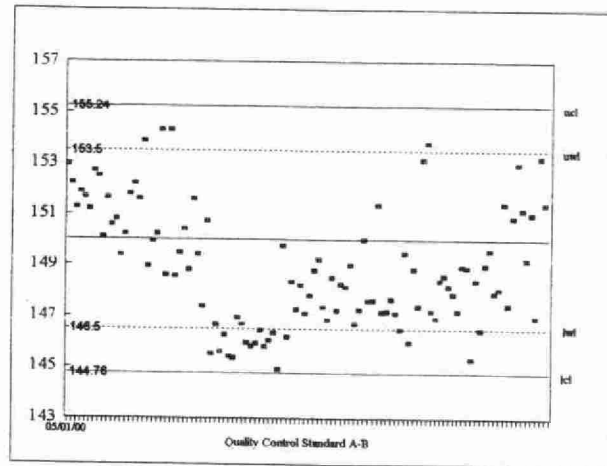
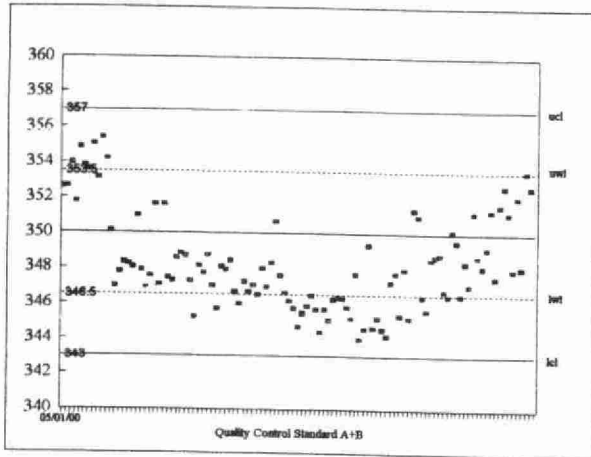
On any given day the calibration is accepted if the calibration control values obtained lie within the ranges:

343	-	357.0	for	A+B
144.76	-	155.24	for	A-B
121.5	-	128.5	for	B+C
72.4	-	77.6	for	B-C
25.82	-	29.18	for	C+D
21.24	-	23.76	for	C-D

DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
60	0 - 50	0.4118	2.0
79	51 - 100	0.5875	0.7
139	101 - 300	0.4307	0.2
22	301 - 1000	1.0247	0.3
300	Overall	0.7951	

Alkalinity , Total Fixed Endpoint (mg/L as CaCO3)
Quality Control Data from 05/01/00 to 19/12/00
E3218



CARBON, DISSOLVED INORGANIC

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/04/78
Method Reference No.	E3370	Reporting Unit	mg/L as C
LIMS Product Code	DCSI3370	Supervisor	P. Wilson
Sample Type/Matrix	Effluent, Industrial Waste, Process Water, Raw Sewage, Drinking Water Ground Water, Leachates, Precipitation, Surface Water		

SAMPLING:

Quantity Required	10 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Dissolved inorganic carbon, which is determined colourimetrically on the supernatant of a settled sample, is converted to carbon dioxide gas by acidification. The gas then passes through a gas-permeable membrane into a weakly-buffered alkaline phenolphthalein solution. The decrease in absorbance of this coloured solution is a measure of the dissolved inorganic carbon content of the sample.

Approximate absorbance: 0.3 at the full scale level.

Dissolved organic carbon, and reactive silicates are determined simultaneously.

INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: air (CO₂-free) supply, dialysis unit. Colourimetric measurement is through a 5.0 cm. light path at 550 nm. Data capture, reduction, and processing via a multi-stage microcomputer system.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.2	Current T value: 1.0
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CALIBRATION:

BL plus 7 standards

CONTROLS:

Calibration	LTBL plus 3 standards, e.g., QCA
Drift	BL, standard and BL every 10 samples

NOTES:

December 1998: The HP data capture/processing system was replaced by Labtronics.

CARBON, DISSOLVED INORGANIC (E3370)

QUALITY CONTROL DATA FROM 07/01/00 TO 18/12/00

Full Scale: to 80.0 mg/L as C

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	68	64.0	63.97	-0.03	0.8540
B:	68	16.0	15.93	-0.07	0.3069
C:	68	4.00	4.08	0.08	0.2052
A+B:		80.0	79.90	-0.10	0.9409
A-B:		48.0	48.04	0.04	0.8727
B+C:		20.0	20.01	0.01	0.4516
B-C:		12.0	11.86	-0.14	0.2622

s.d.(AB)

S(between runs): 0.62

Sw(within run): 0.63

S/Sw:1.0

s.d.(BC)

S(between runs): 0.23

Sw(within run): 0.18

S/Sw:1.3

The calibration is accepted if the calibration control values obtained lie within the ranges:

77.60	-	82.40	for	A+B
46.40	-	49.60	for	A-B
18.83	-	21.17	for	B+C
11.22	-	12.88	for	B-C

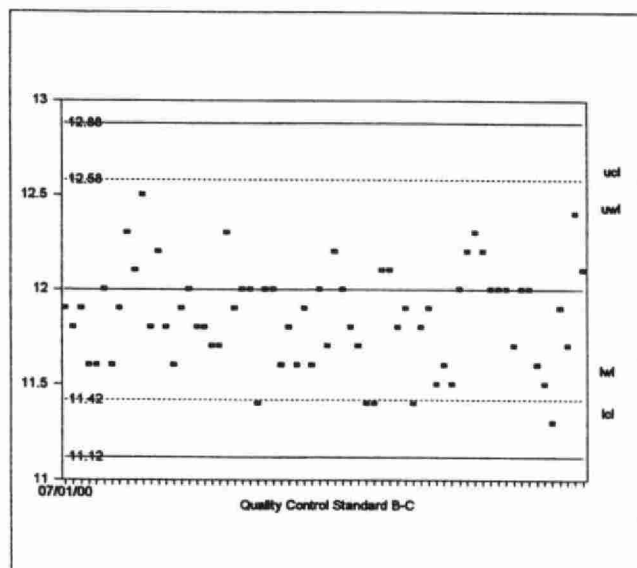
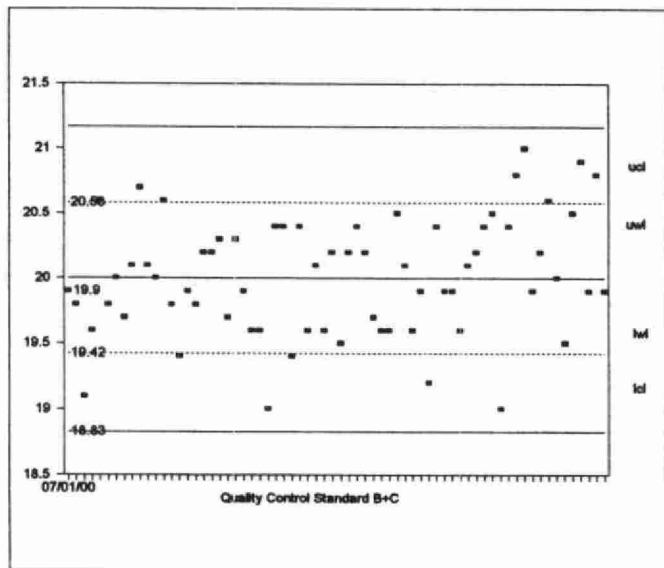
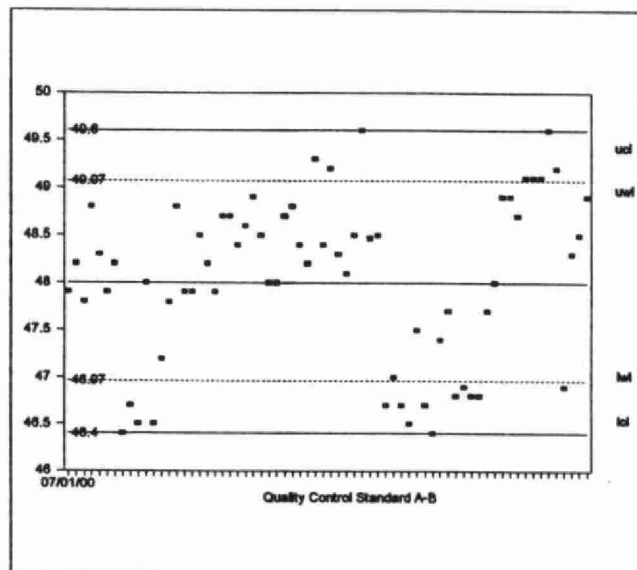
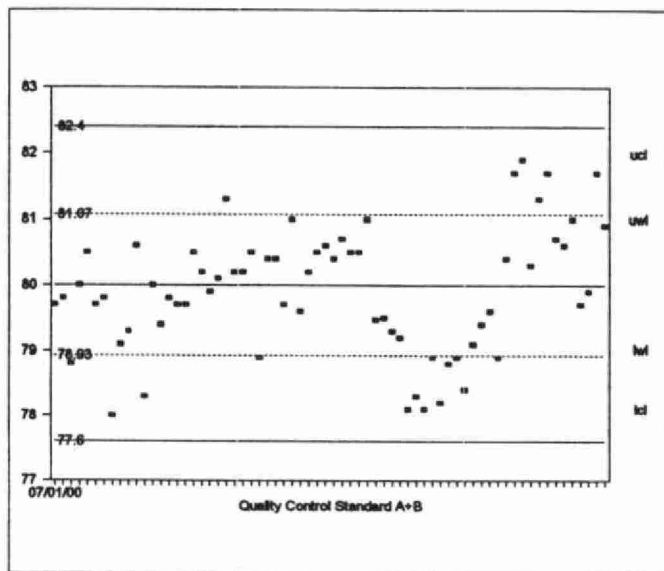
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
68	0.00 - 16.0	0.3217	9.1
76	16.1 - 40.0	0.4355	1.9
43	40.1-80.0	0.9157	1.7
187	Overall	0.5545	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	68	0.0559	0.2427

CARBON, DISSOLVED INORGANIC (mg/L as C)
QUALITY CONTROL DATA FROM 07/01/00 TO 18/12/00
E3370



CARBON, DISSOLVED ORGANIC

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/04/78
Method Reference No.	E3370	Reporting Unit	mg/L as C
LIMS Product Code	DCSI3370	Supervisor	P. Wilson
Sample Type/Matrix	Effluent, Industrial Waste, Process Water, Raw Sewage, Drinking Water Ground Water, Leachates, Precipitation, Surface Water		

SAMPLING:

Quantity Required	10 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Using an automated system, the supernatant from a settled sample is acidified and flushed with nitrogen gas to remove inorganic carbon. Organic carbon is then oxidized to carbon dioxide gas by exposure to ultra-violet light (UV) in acid-persulphate media. The gas then passes through a gas-permeable membrane into a weakly-buffered alkaline phenolphthalein solution. The decrease in absorbance of this coloured solution is a measure of the dissolved organic carbon content of the sample.

Approximate absorbance: 0.3 at the full scale level.

Dissolved inorganic carbon, and reactive silicates are determined simultaneously.

INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: nitrogen and air (CO₂ -free) supplies with flow controls, dialysis unit, UV digester. Colourimetric measurement is through a 5.0 cm. light path at 550 nm. Data capture, reduction, and processing via a multi-stage microcomputer system.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.1	Current T value: 0.5
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CALIBRATION:

BL plus 7 standards

CONTROLS:

Calibration	LTBL plus 3 standards, e.g., QCA
Drift	BL , standard and BL every 10 samples

NOTES:

December 1998: The HP data capture/processing system was replaced by Labtronics.

CARBON, DISSOLVED ORGANIC (E3370)

QUALITY CONTROL DATA FROM 07/01/00 TO 18/12/00

Full Scale: to 20.0 mg/L as C

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	68	16.0	15.91	-0.090	0.1394
B:	68	4.00	4.00	-0.003	0.1146
C:	68	1.00	0.99	-0.010	0.1024
A+B:		20.0	19.91	-0.093	0.1818
A-B:		12.0	11.91	-0.087	0.1795
B+C:		5.00	4.99	-0.013	0.1795
B-C:		3.00	3.01	0.007	0.1226

s.d.(AB) S(between runs): 0.1276

Sw(within run): 0.1269

S/Sw: 1.0

s.d.(BC) S(between runs): 0.1087

Sw(within run): 0.0867

S/Sw: 1.3

The calibration is accepted if the calibration control values obtained lie within the ranges:

19.44	-	20.56	for	A+B
11.58	-	12.42	for	A-B
4.56	-	5.44	for	B+C
2.67	-	3.33	for	B-C

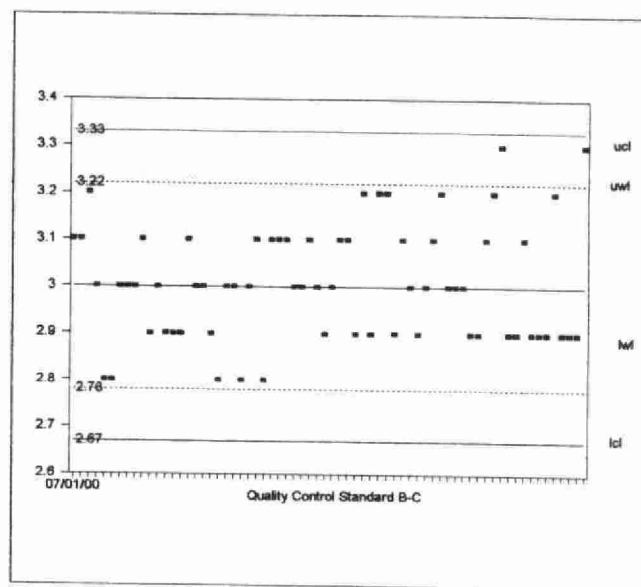
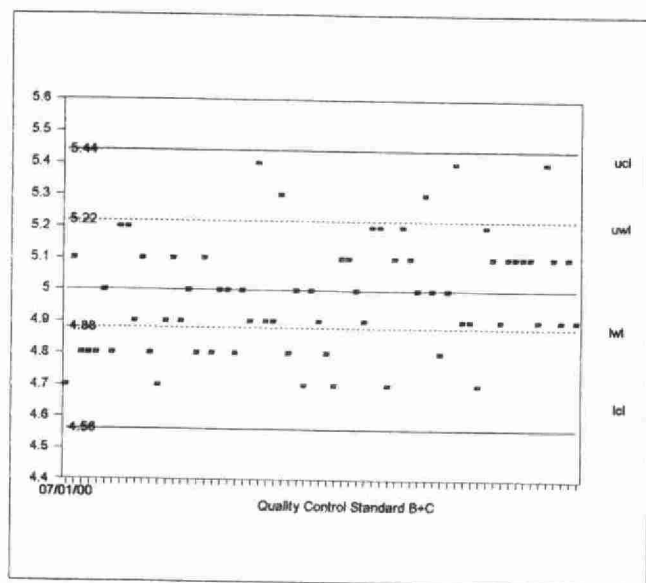
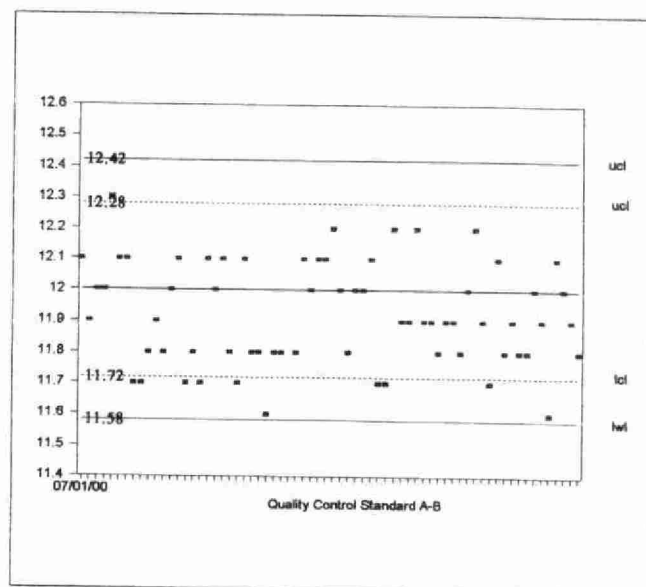
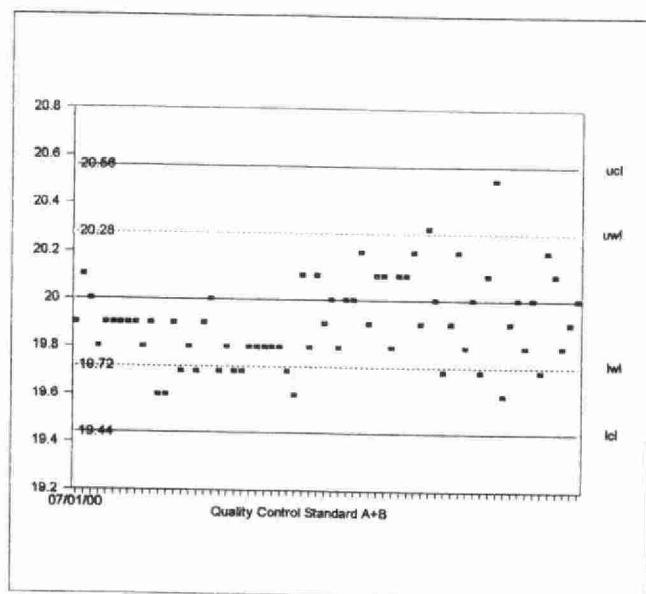
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
58	0.0 - 2.0	0.1569	11.3
64	2.1 - 4.0	0.1700	6.1
51	4.1 - 10.0	0.1502	2.5
16	10.1 - 20.0	0.2450	1.6
189	Overall	0.1688	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	68	-0.0088	0.1116

CARBON. DISSOLVED ORGANIC (mg/L as C)
QUALITY CONTROL DATA FROM 07/01/00 TO 18/12/00
E3370



CHLORIDE

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/04/78
Method Reference No.	E3004	Reporting Unit	$\mu\text{g}/\text{m}^3$ as Cl
LIMS Product Code	ANION3004	Supervisor	P. Wilson
Sample Type/Matrix	Air; HiVol Glass Fibre, Quartz and Polyflon, Other Filters and Puff		

SAMPLING:

Quantity Required	3/4" or 1.9cm strip from 8"x10" filter
Container	50 mL polypropylene tube

SAMPLING PREPARATION:

A 3/4" filter strip is cut in pieces and deposited into a 50 mL polypropylene tube. 50 mL of Pure-Water is added to the tube. The tube is placed on a horizontal shaker for approximately 1 hour. The supernatant is then filtered into a 15 mL plastic tube and analysed.

ANALYTICAL PROCEDURE:

Chloride is separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of sodium bicarbonate and sodium carbonate and a conductivity detector. The concentration of chloride (mg/L) is determined by the comparison of the analyte peak area count to that of a series of standards. The analyte result is corrected for the filter blank before the final calculation. The result is reported as $\mu\text{g}/\text{m}^3$ as Cl.

Nitrate and sulphate are determined simultaneously.

INSTRUMENTATION:

Horizontal Shaker, ion chromatographic system plus a PC with ChromPerfect software and DT2804 card for automated sample injection, timing, and data processing.

REPORTING:

Maximum Significant Figures: 2	Current W value: $0.1 \mu\text{g}/\text{m}^3$	Current T value: $0.5 \mu\text{g}/\text{m}^3$
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CALIBRATION:

6 standards

CONTROLS:

Calibration	MB, IS(n), CS1, and CS2
Drift	Duplicate plus 2 standards approximately every 20 samples

CHLORIDE cont'd

NOTES:

To convert unit from mg/L to $\mu\text{g}/\text{m}^3$, the final concentration of Cl in mg/L is multiplied by the following formula:

$$\text{Result (mg/L)} \times 50\text{mL} \times (63/6.75) / \text{air volume} = \mu\text{g}/\text{m}^3$$

Where: 63 is the area of the filter exposed and 6.75 is the sample aliquot area in square inch.

CHLORIDE (E3004)

QUALITY CONTROL DATA FOR 2000

Analytical Range: to 14.31 $\mu\text{g}/\text{m}^3$

DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
83	0.0 - 2.86	0.027	12.0
3	2.89 - 7.15	0.058	1.7
0	7.18 - 14.31	N.A.	N.A.
86	Overall	0.029	

CHLORIDE

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/01/86
Method Reference No.	E3013	Reporting Unit	µg/g as Cl
LIMS Product Code	ANION3013, CL3013	Supervisor	P. Wilson
Sample Type/Matrix	Soil and Sediment		

SAMPLING:

Quantity Required	20 g
Container	glass or plastic

SAMPLING PREPARATION:

A 3.0 g sample air dried, sieved soil or air dried sieved and ground sediment is placed in a 50 mL centrifuge tube and shaken with 30 mL Pure-DW for 1 hour on a shaker. Samples are centrifuged, membrane filtered and analyzed for chloride and sulphate by ion chromatography.

ANALYTICAL PROCEDURE:

Chloride is separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of sodium bicarbonate and sodium carbonate and a conductivity detector. The concentration of chloride (mg/L) is determined by the comparison of the analyte peak area count to that of a series of standards. The result is reported as µg/g as Cl. Sulphate is determined simultaneously.

INSTRUMENTATION:

Horizontal Shaker, ion chromatographic system plus a PC with ChromPerfect software and DT2804 card for automated sample injection, timing, and data processing.

REPORTING:

Maximum Significant Figures: 2	Current W value: 0.5 µg/g	Current T value: 2.5 µg/g
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CALIBRATION:

8 standards

CONTROLS:

Calibration	MB, IS(n), CS1, and CS2
Drift	Duplicate plus 2 standards approximately every 20 samples

CHLORIDE (E3013)

QUALITY CONTROL DATA FOR 2000

Analytical Range: to 500 $\mu\text{g/g}$

DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
83	0.0 - 100	1.5668	9.1
9	101 - 250	2.5471	1.4
2	251 - 500	4.0305	1.5
94	Overall	1.7705	

CHLORIDE

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/05/75
Method Reference No.	E3016	Reporting Unit	mg/L as Cl
LIMS Product Code	CL3016	Supervisor	P. Wilson
Sample Type/Matrix	Effluent, Industrial Waste, Process Water, Raw Sewage, Drinking Water, Ground Water, Leachate, Surface Water		

SAMPLING:

Quantity Required:	10 mL
Container:	Plastic

ANALYTICAL PROCEDURE:

Chloride ions are combined with mercuric thiocyanate releasing thiocyanate quantitatively. Thiocyanate then reacts with ferric ions to produce ferric thiocyanate (red), and the absorbance of the latter is measured colourimetrically.

Approximate absorbance: 0.5 at the full scale level.

INSTRUMENTATION:

Basic automated modular continuous flow system with colourimetric measurement through a 1.5 cm light path at 480 nm.

Data capture and processing via a multistage microcomputer system.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.2	Current T value: 1
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CALIBRATION:

BL plus 12 standards

CONTROLS:

Calibration:	LTBL plus 3 standards, e.g. QCA
Drift:	BL and standard after every 12 samples

NOTES:

April 1998: The HP data capture/processing system was replaced by Labtronics. Two additional Calibration standards were added at the low end of the curve.

CHLORIDE (E3016)

QUALITY CONTROL DATA FROM 07/01/00 TO 28/12/00

Full Scale: to 100 mg/L as Cl

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	78	75.00	75.20	0.20	0.2972
B:	78	25.00	25.12	0.12	0.1330
C:	78	5.00	4.90	-0.10	0.0579
A+B:	78	100.00	100.32	0.32	0.3557
A-B:	78	50.00	50.09	0.09	0.2924
B+C:	78	30.00	30.01	0.01	0.1610
B-C:	78	20.00	20.22	0.22	0.1272

s.d.(AB) S(between runs):0.23
s.d.(BC) S(between runs):0.10

Sw(within run): 0.21 S/Sw: 1.1
Sw(within run): 0.09 S/Sw: 1.1

The calibration is accepted if the calibration control values obtained lie within the ranges:

98.7 - 101.3 for A+B
49.0 - 51.0 for A-B
29.3 - 30.7 for B+C
19.5 - 20.5 for B-C

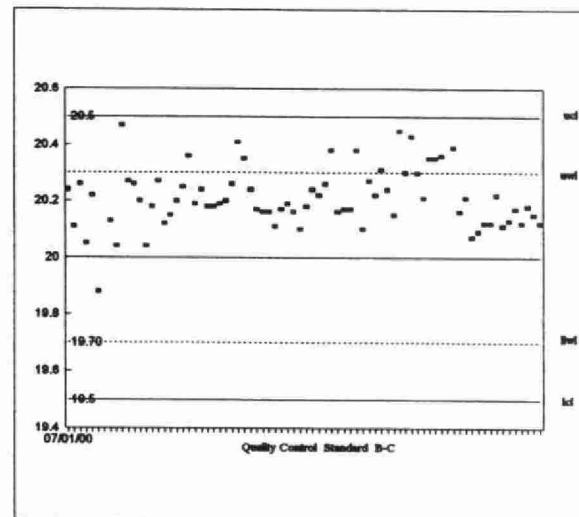
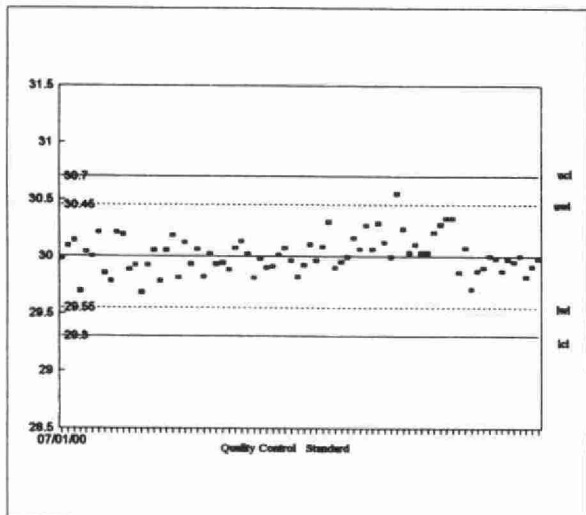
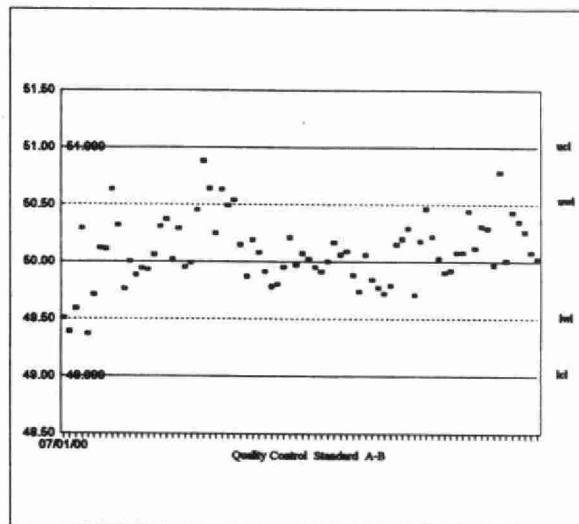
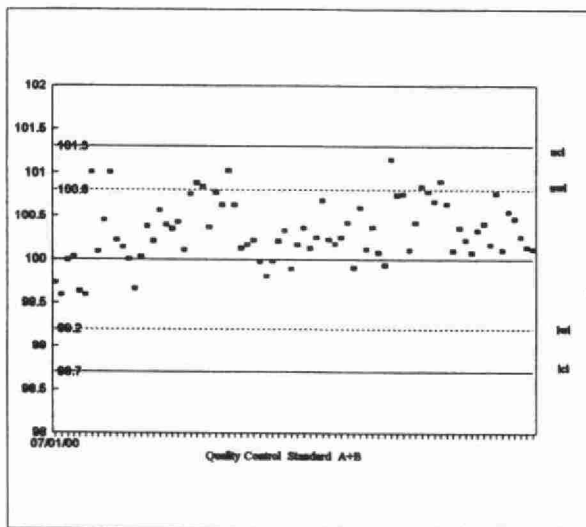
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
58	0.00 - 10.0	0.1408	3.4
54	10.1 - 20.0	0.1125	0.7
67	20.1 - 50.0	0.2707	0.9
32	50.1 - 100	0.3378	0.5
211	Overall	0.2220	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	78	0.044	0.0342

Chloride (mg/L as Cl)
Quality Control Data From 07/01/00 To 28/12/00
E3016A



CHLOROPHYLL

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/04/75
Method Reference No	E3169	Reporting Unit	µg/L
LIMS Product Code	CHL3169	Supervisor	P. Wilson
Sample Type/Matrix	Effluent, Drinking Water, Surface Water		

SAMPLING:

Quantity Required	1000 mL for clear samples; 500 mL if visibly green
Container	Glass or plastic
Other	In the field a sample is filtered through a nylon filter. The filter is folded and then placed between two membrane filter-support pads, and the package is enclosed in a plastic dish labelled with the sample number and sample volume filtered, the dish is kept in the dark or wrapped in aluminum foil, and shipped immediately, or kept frozen.

ANALYTICAL PROCEDURE:

Using an IBM clone microcomputer-controlled, automated spectrophotometer, two scans are developed with absorbance measurements at 630, 645, and 663 nm for the first scans; the minimum absorbance value between 710 and 750 nm (readings at 5 nm intervals) is utilized as a turbidity correction. Chlorophyll "a" and "b" are calculated from this scan. After automated acidification, the second scan is obtained from the wavelength 665 nm for correcting chlorophyll "a" measurement. SCOR-UNESCO equations are used for all chlorophyll calculations.

INSTRUMENTATION:

- Automated modular continuous flow scanning spectrophotometer system
- Microcomputer system for control of sampling, timing and data processing (i.e. data capture, calculations and transfer of results to LIMS)

REPORTING:

Chlorophyll a; corrected	Maximum Significant	Current W value: 1.0	Current T value: 5.0
Chlorophyll a; total	Figures: 3	Current W value: 0.2	Current T value: 1.0
Chlorophyll b; total		Current W value: 0.1	Current T value: 0.5

CONTROLS:

Calibration	LTBL plus 2 "standards", e.g.QCA
Drift	"standard", BL every 20 samples

NOTES:

"Standards" are prepared from chlorophyll "a" and "b", but the materials are neither analytical grade nor are their solutions stable. Thus calibration controls are based on measured averages.

CHLOROPHYLL cont'd

NOTES (cont):

Jan 1999, the microcomputer system was replaced with a 486 P.C. The software written in PET BASIC was replaced by software written in CLIPPER.

May 2000 the Bechman DU7 spectrophotometer was replaced with a Bechman DU600 instrument.

CHLOROPHYLL "a" (E3169)

QUALITY CONTROL DATA FROM 11/01/00 TO 21/12/00

Reporting Unit: µg/L

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	35	3.0	3.02	0.02	0.1338
B:	35	1.0	1.04	0.04	0.1142
A+B:		4.0	4.06	0.06	0.2188
A-B:		2.0	1.98	-0.02	0.1184

s.d.(AB) S(between runs): 0.12 Sw(within run): 0.08 S/Sw: 1.5

The calibration is accepted if the calibration control values obtained lie within the ranges:

3.6 - 4.4 for A+B
1.7 - 2.3 for A-B

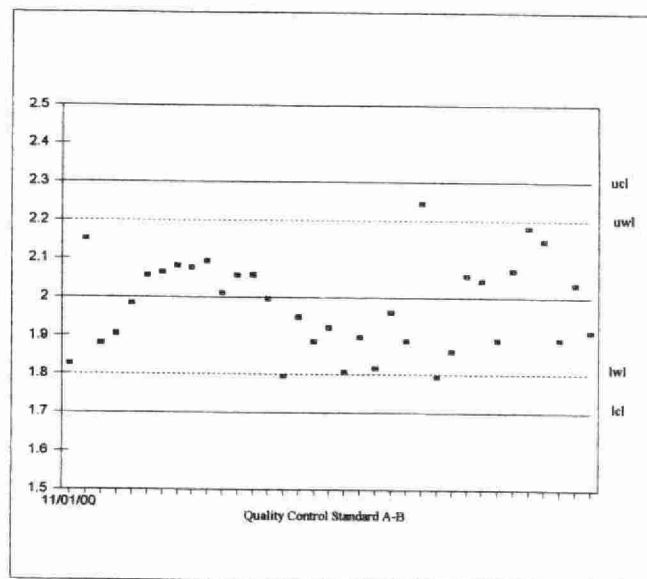
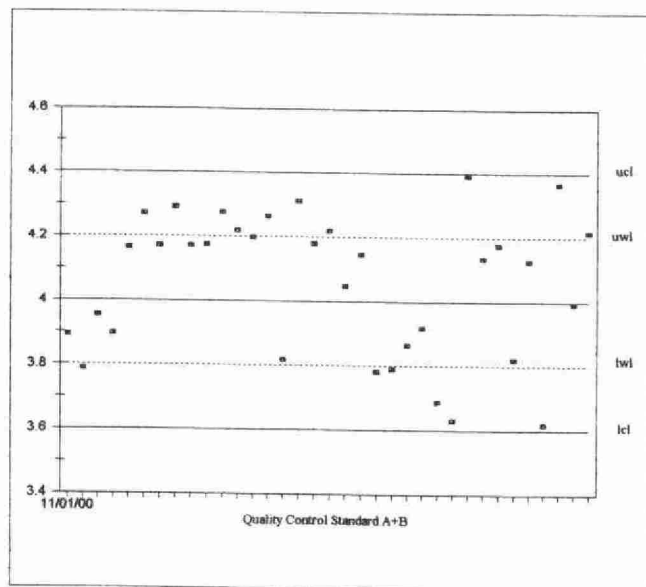
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
33	0 - 5.0	0.1605	16.3
2	5.1 - 10.0	N.A.	N.A.
0	10.1 - 25.0	N.A.	N.A.
35	Overall	0.1963	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	35	0.1107	0.0662
Filtered Blank	35	0.1100	0.0843

CHLOROPHYLL "a" (ug/L)
QUALITY CONTROL DATA FROM 11/01/00 TO 21/12/00
E3169



CHLOROPHYLL "a", ACIDIFIED (E3169)

QUALITY CONTROL DATA FROM 11/01/00 TO 21/12/00

Reporting Unit: µg/L

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	35	2.4	2.61	0.21	0.2879
B:	35	0.8	0.87	0.07	0.2266
A+B:		3.2	3.48	0.28	0.4777
A-B:		1.6	1.74	0.14	0.2005

s.d.(AB) S(between runs): .26 Sw(within run): 0.14 S/Sw: 1.8

The calibration is accepted if the calibration control values obtained lie within the ranges:

2.4 - 4.0 for A+B
1.0 - 2.2 for A-B

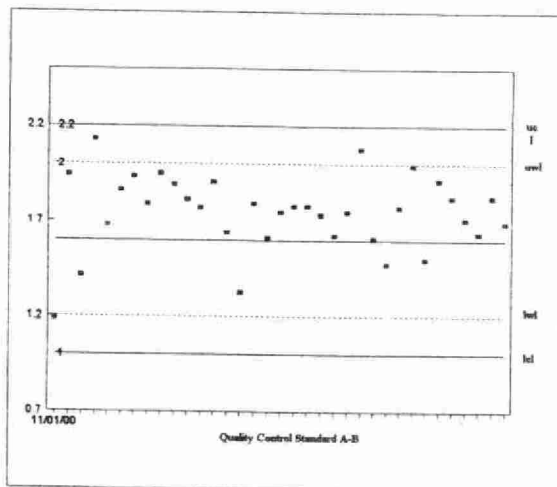
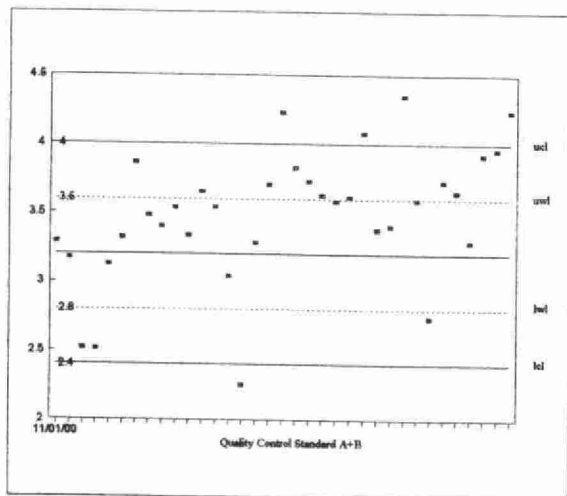
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
30	-0.5 - 1.0	0.3282	130.8
3	1.1 - 2.0	0.3644	137.2
4	2.1 - 5.0	0.8153	61.3
0	5.1 - 10.0	N.A.	N.A.
0	10.1 - 100	N.A.	N.A.
37	Overall	0.4484	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	35	0.0549	0.1946
Filtered Blank	35	0.0180	0.1861

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CHLOROPHYLL "b" (E3169)

QUALITY CONTROL DATA FROM 11/01/00 TO 21/12/00

Reporting Unit: µg/L

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	35	3.0	2.98	-0.02	0.1318
B:	35	1.0	1.07	0.07	0.1230
A+B:		4.0	4.05	0.05	0.2168
A-B:		2.0	1.91	-0.09	0.1343

s.d.(AB)

S(between runs):0.128

Sw(within run): 0.095

S/Sw: 1.3

The calibration is accepted if the calibration control values obtained lie within the ranges:

3.6 - 4.4 for A+B
1.7 - 2.3 for A-B

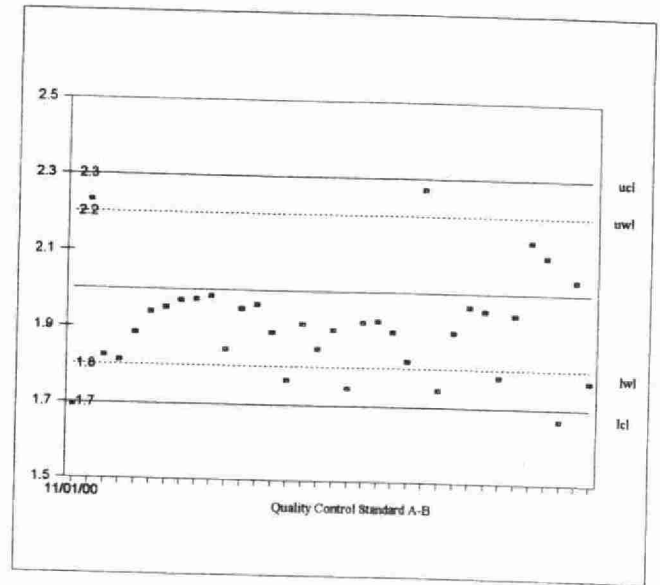
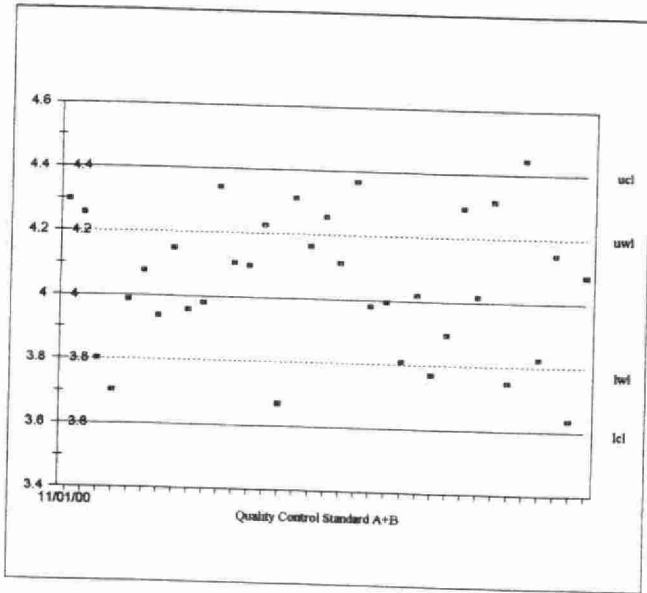
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
30	0 - 1.0	0.2102	38.7
5	1.1 - 2.0	0.2862	20.8
1	2.1 - 5.0	N.A.	N.A.
36	Overall	0.2742	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	35	0.1627	0.1121
Filtered Blank	35	0.1719	0.1293

CHLOROPHYLL "b" ($\mu\text{g/L}$)
QUALITY CONTROL DATA FROM 11/01/00 TO 21/12/00
E3169



COLOUR, TRUE

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	13/03/84
Method Reference No.	E3219	Reporting Unit	TCU
LIMS Product Code	COL3219	Supervisor	P. Wilson
Sample Type/Matrix	Effluent, Industrial Waste, Drinking Water, Ground Water, Leachate, Precipitation, Surface Water		

SAMPLING:

Quantity Required	50 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

True colour is measured colourimetrically on the supernatant of a settled sample in a system calibrated with acidified chloroplatinate standards. The sample stream is measured using a broadband blue filter. Residual turbidity effects are suppressed by using a broadband red filter and increased path length in the reference stream.

Approximate absorbance: 0.3 at the full scale level.

INSTRUMENTATION:

Basic automated modular continuous flow system. Colour measurement is through a 3.0 cm. light path using a broadband filter (400-450 nm). Turbidity measurement is through a 5.0 cm. light path using a different broadband filter (660-740 nm). Data capture, and processing via a multi-stage microcomputer system.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.2	Current T value: 1
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CALIBRATION:

BL plus 6 standards

CONTROLS:

Calibration	LTBL plus 2 standards, e.g. QCA
Drift	BL and standard after every 10 samples

NOTES:

The HP data capture/processing system was replaced by Labtronics in November 1998.

COLOUR, TRUE (E3219)

QUALITY CONTROL DATA FROM 07/01/00 TO 22/12/00

Analytical Range: to 100 TCU

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	37	70.0	70.27	0.27	0.4380
B:	37	25.0	25.51	0.51	0.3590
C:	37	7.5	7.27	-0.23	0.2511
A+B:	37	95.0	95.78	0.78	0.6457
A-B:	37	45.0	44.76	-0.24	0.4738
B+C:	37	32.5	32.78	0.28	0.4990
B-C:	37	17.5	18.24	0.74	0.3671

s.d.(AB) S(between runs): 0.40

Sw(within run): 0.34

S/Sw: 1.2

s.d.(BC) S(between runs): 0.31

Sw(within run): 0.26

S/Sw: 1.2

On any given day the calibration is accepted if the calibration control values obtained lie within the ranges:

92.2	-	97.8	for	A+B
42.9	-	47.1	for	A-B
30.6	-	34.3	for	B+C
16.1	-	18.9	for	B-C

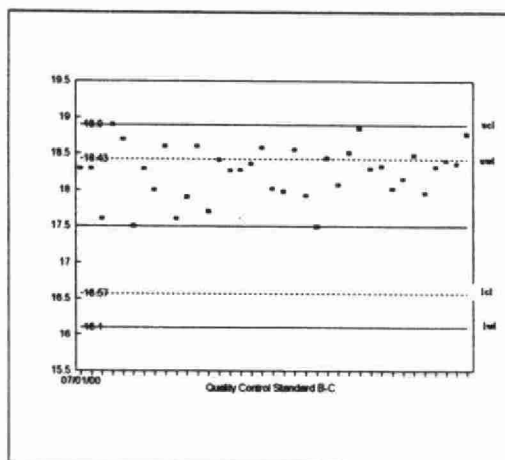
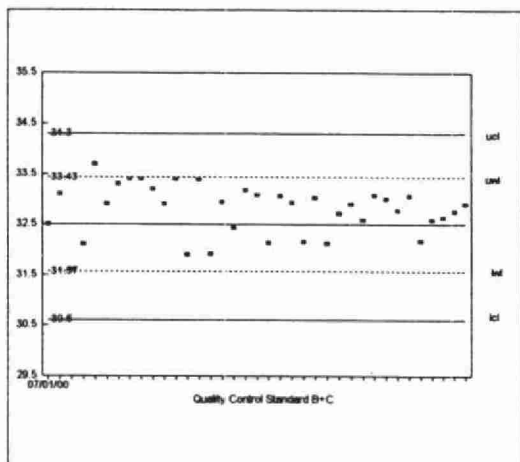
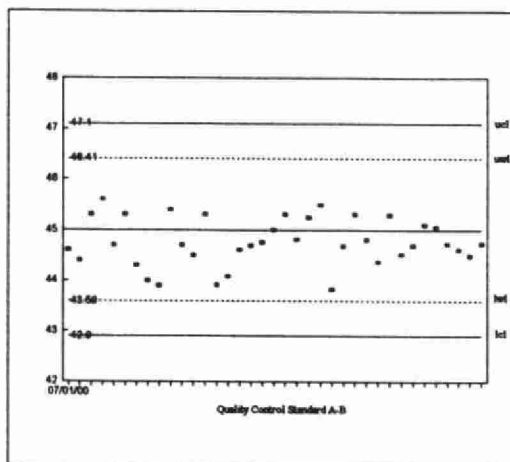
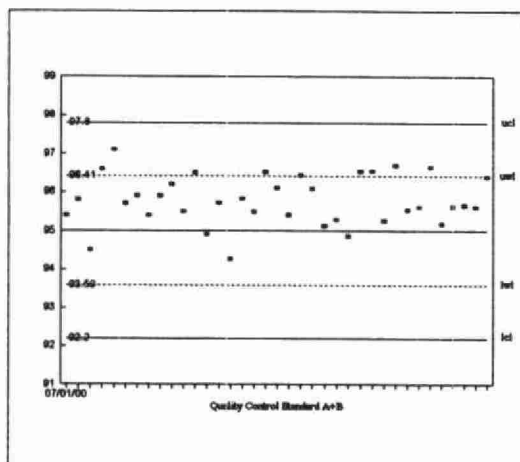
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
86	0.0 - 10.0	0.3223	14.9
8	10.1 - 20.0	0.2085	1.7
15	20.1 - 50.0	0.3691	1.3
1	50.1 - 100.0	N.A.	N.A.
110	Overall	0.3215	

OTHER CHECKS:

	n	Data Mean	Standard (1) Deviation
Long Term Blank	37	-0.5049	0.3387

Colour, True (TCU)
 Quality Control Data from 07/01/00 to 22/12/00
 E3219



CONDUCTIVITY

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced:	01/04/74
Method Reference No:	E3218	Reporting Units:	$\mu\text{S/cm}$ at 25°C
LIMS Product Code:	PHALCO3218,CONDPH3218	Supervisor:	P. Wilson
Sample Type/Matrix:	Sludge, Effluent, Industrial Waste, Raw Sewage, Drinking Water, Ground Water, Leachate, Precipitation, Surface Water		

SAMPLING:

Quantity Required:	50 mL
Container:	Glass or plastic

ANALYTICAL PROCEDURE:

After equilibration at 25°C, the conductivity of the sample is measured. pH, and total fixed endpoint alkalinity are determined simultaneously.

INSTRUMENTATION:

Automated modular continual flow conductivity system comprising of a sampler and conductivity meter with cell plus microcomputer control and data processing software.

REPORTING:

Maximum Significant Figures: 3	Current W value: 1	Current T value: 5
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CONTROLS:

Calibration:	LTBL plus 4 standards, e.g. QCA
Drift:	In run standards throughout the run (tap water diluted to 50% V/V)

CONDUCTIVITY (E3218)

QUALITY CONTROL DATA FROM 05/01/00 TO 19/12/00

Analytical Range: to 2000 $\mu\text{S/cm}$

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	111	1413	1411.2	-1.8	3.7516
B:	111	718	717.2	-0.8	2.3977
C:	111	147	148.0	1.0	0.5890
D:	111	37.1	38.2	1.1	0.2828
A+B:		2131	2128.4	-2.6	5.3260
A-B:		695	694.0	-1.0	3.3587
B+C:		865	865.1	0.1	2.6722
B-C:		571	569.2	-1.8	2.2474
C+D:		184.1	186.2	2.1	0.7037
C-D:		109.9	119.7	9.8	0.5988

s.d.(AB) S(between runs): 3.15

Sw(within run): 2.38

S/Sw: 1.3

s.d.(BC) S(between runs): 1.75

Sw(within run): 1.59

S/Sw: 1.1

s.d.(CD) S(between runs): 0.46

Sw(within run): 0.42

S/Sw: 1.1

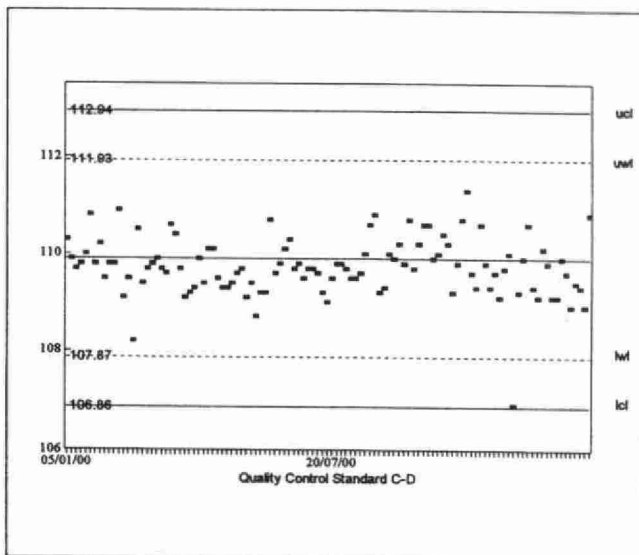
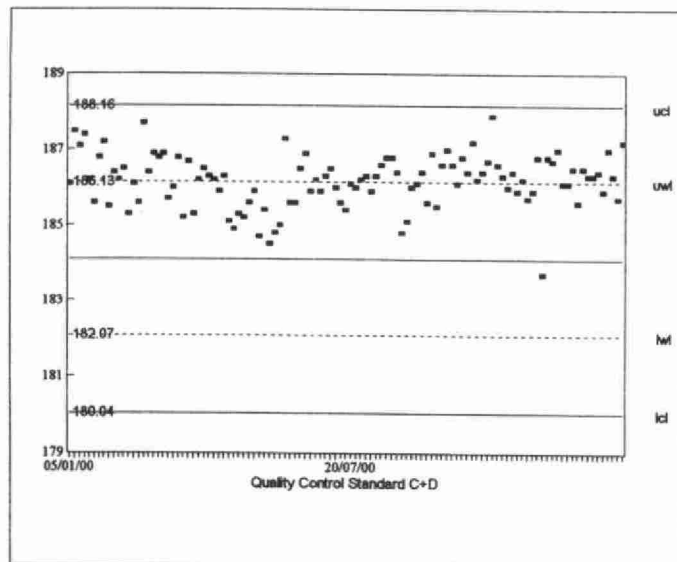
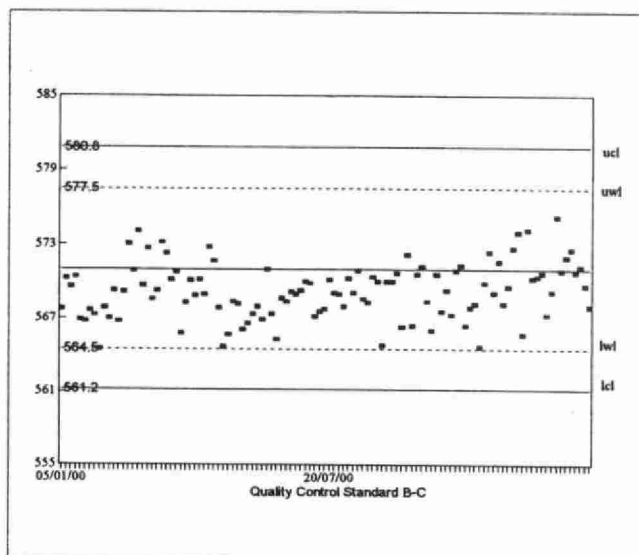
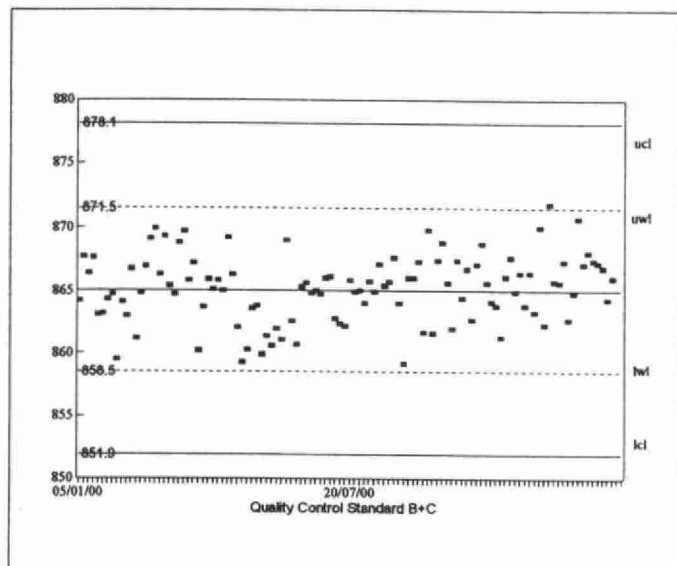
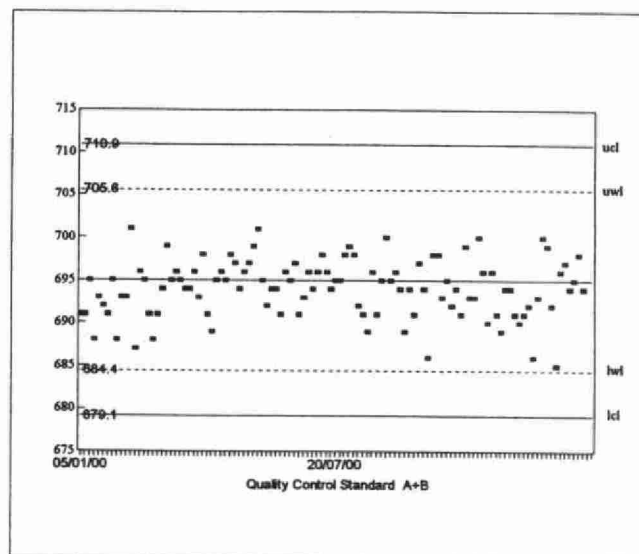
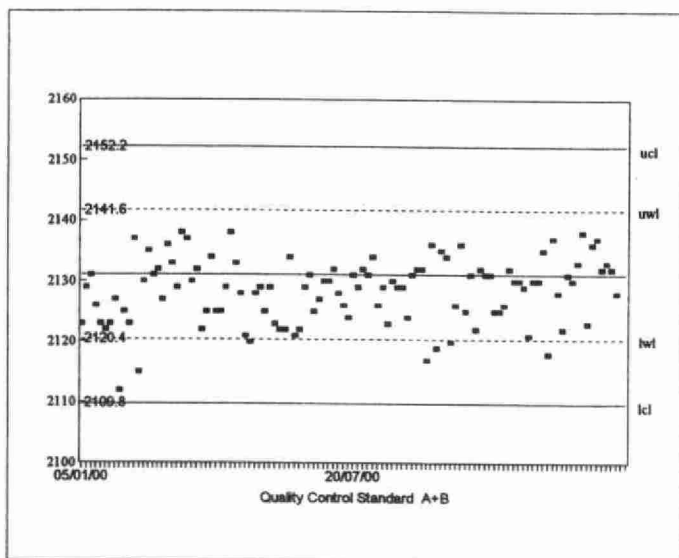
On any given day the calibration is accepted if the calibration control values obtained lie within the ranges:

2109.8	-	2152.2	for	A+B
679.1	-	710.9	for	A-B
851.9	-	878.1	for	B+C
561.2	-	580.8	for	B-C
180.04	-	188.16	for	C+D
106.86	-	112.94	for	C-D

DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
145	0 - 400	1.8574	1.0
98	401 - 1000	3.3685	0.5
33	1001 - 2000	3.5011	0.3
33	2001 - 10000	16.5145	0.5
309	Overall	5.9710	

Conductivity ($\mu\text{S}/\text{cm}$)
 Quality Control Data from 05/01/00 to 19/12/00
 E3218



CYANIDE, FREE

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/01/98
Method Reference No.	E3015	Reporting Unit	Aqueous: mg/L as CN^- Solid: $\mu\text{g/g}$ as CN^-
LIMS Product Code	CNF3015	Supervisor	P. Wilson
Sample Type/Matrix	Aqueous: Surface Water, Drinking Water, Ground Water, Raw Sewage & Effluent, Industrial Effluent. Solid: Soil, Sediment, Vegetation, Dried Sludge, Industrial Waste		

SAMPLING:

Quantity Required:	Aqueous: 500 mL + 10 drops of 50% w/v NaOH Solid : 5 g, minimum
Container:	Glass or plastic

ANALYTICAL PROCEDURE:

Free cyanides are the simple and weakly dissociable cyanides which form HCN upon acidification to pH4.0 (such as HCN and KCN). The automated determination of free cyanide exposes the sample to distillation which isolates HCN under specific acidic conditions. A zinc sulphate solution is included which eliminates interference from complexed iron cyanides. Cyanide is determined colourimetrically by the reaction of cyanide with chloramine -T to form cyanogen chloride which further reacts with a combination of barbituric acid and isonicotinic acid to form a highly coloured coupling product, which is measured at 600 nm.

Aqueous samples are introduced directly to the continuous flow system by an autosampler. Solid samples are extracted in a sodium hydroxide solution with mechanical shaking for 6 to 8 hours and then centrifuged. The supernatant is decanted, diluted if necessary to eliminate interference from colour and introduced to the continuous flow system by the autosampler. Solid samples are not dried or ground, but weighed and extracted as received, to prevent the loss of simple cyanides. If the sample is wet, results are reported as $\mu\text{g/g}$ wet and moisture content is reported by a separate method.

INSTRUMENTATION:

Skalar automated segmented flow colourimetric system, measurement through a 500 mm light path at 600 nm.

Skalar data capture and data processing software with computer system.

REPORTING:

Maximum Significant Figures: 3 or the nearest W	Current W value: 0.001 mg/L 0.01 $\mu\text{g/g}$	Current T value: 0.005mg/L 0.05 $\mu\text{g/g}$
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CALIBRATION:

BL plus 6 standards (S0 to S5)

CONTROLS:

Calibration:	LTB plus 2 standards , e.g. QCA
Drift:	BL and check standards

CYANIDE, FREE (E3015)

QUALITY CONTROL DATA FROM 08/02/00 TO 30/10/00

Laboratory Unit: Colourimetry

Full Scale: to 0.2 mg/L as CN

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	10	0.15	0.1496	-0.0004	0.0013
B:	10	0.02	0.0189	-0.0011	0.0010
A+B:		0.17	0.1686	-0.0014	0.0019
A-B:		0.13	0.1307	0.0007	0.0012

s.d.(AB)

S(between runs): 0.0011

Sw(within run): 0.0008

S/Sw: 1.4

The calibration is accepted if the calibration control values obtained lie within the ranges:

0.154 - 0.186 for A+B

0.118 - 0.142 for A-B

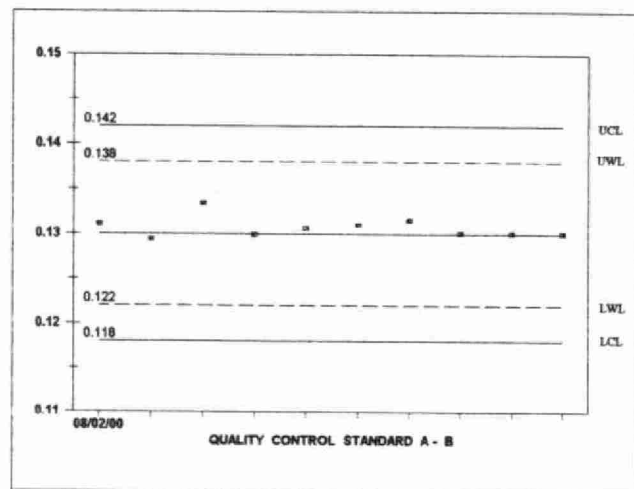
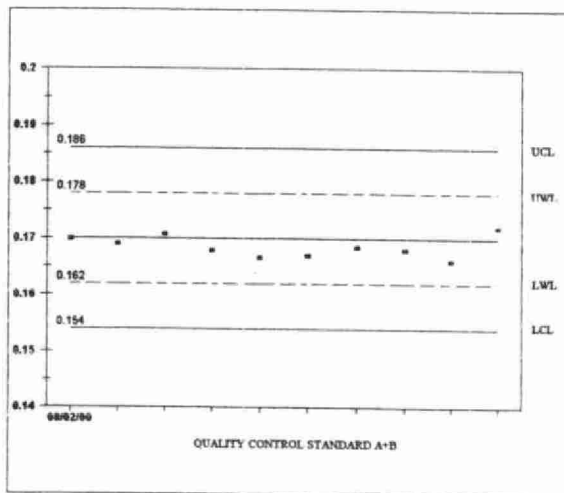
REFERENCE MATERIAL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
KCN:	10	0.10	0.0909	0.0091	0.0127
FeCN:	10	0.10	0.0013	0.0987	0.0007

DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
18	0 - 0.020	0.0007	11.5
1	0.021 - 0.040	N.A.	N.A.
5	0.041 - 0.100	0.0005	0.8
0	0.101 - 0.200	N.A.	N.A.
24	Overall	0.0008	

CYANIDE FREE (mg/L as CN)
QUALITY CONTROL DATA FROM 08/02/00 TO 30/10/00
E3015



CYANIDE, TOTAL

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/01/98
Method Reference No.	E3015	Reporting Unit	Aqueous: mg/L as CN^- Solid: $\mu\text{g/g}$ as CN^-
LIMS Product Code	CN3015	Supervisor	P. Wilson
Sample Type/Matrix	Aqueous: Surface Water, Drinking Water, Ground Water, Raw Sewage & Effluent, Industrial Effluent. Solid: Soil, Sediment, Vegetation, Dried Sludge, Industrial Waste		

SAMPLING:

Quantity Required:	Aqueous: 500 mL + 10 drops of 50% w/v NaOH Solid : 5 g, minimum
Container:	Glass or plastic

ANALYTICAL PROCEDURE:

Total cyanides include free, simple (HCN , KCN) and weakly dissociable cyanides ($\text{Ni}(\text{CN})_4$) as well as those complexed cyanides that decompose to form free cyanides that distill out as HCN in an acidic environment. The automated determination of total cyanide exposes the sample to ultraviolet radiation to break down organic metallic and alkali-complexed cyanide compounds to free cyanide. The distillation step isolates HCN under specific acidic conditions. The sequential combination of UV digestion plus distillation yields the measurement of "total cyanide". Cyanide is measured colourimetrically by the reaction of cyanide with chloramine -T to form cyanogen chloride which further reacts with a combination of barbituric acid and isonicotinic acid to form a highly coloured coupling product, which is measured at 600 nm.

Aqueous samples are introduced directly to the continuous flow system from an autosampler. Solid samples are extracted in a sodium hydroxide solution with mechanical shaking for 6 to 8 hours, then centrifuged. The supernatant is then decanted, diluted if necessary to eliminate interference from colour and introduced to the continuous flow system by the autosampler. Solid samples are not dried or ground, but weighed and extracted as received, to prevent the loss of simple cyanides. If the sample is wet, results are reported as $\mu\text{g/g}$ wet and moisture content is reported by a separate method.

INSTRUMENTATION:

Skalar automated segmented flow colourimetric system, measurement through a 500 mm light path at 600 nm. Skalar data capture and data processing software with computer system.

REPORTING:

Maximum Significant Figures: 3 or the nearest W	Current W value: 0.001 mg/L 0.01 $\mu\text{g/g}$	Current T value: 0.005mg/L 0.05 $\mu\text{g/g}$
---	---	--

CALIBRATION:

BL plus 6 standards (S0 to S5)

CONTROLS:

Calibration:	LTB plus 2 standards , e.g. QCA
Drift:	BL and check standards

CYANIDE, TOTAL (E3015)

QUALITY CONTROL DATA FROM 08/02/00 TO 22/12/00

Laboratory Unit: Colourimetry

Full Scale: to 0.2 mg/L as CN

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	30	0.15	0.1498	-0.0002	0.0012
B:	30	0.02	0.0195	-0.0005	0.0012
A+B:		0.17	0.1693	-0.0007	0.0020
A-B:		0.13	0.1302	0.0002	0.0013

s.d.(AB)

S(between runs): 0.0012

Sw(within run): 0.0009

S/Sw: 1.3

The calibration is accepted if the calibration control values obtained lie within the ranges:

0.154 - 0.186 for A+B

0.118 - 0.142 for A-B

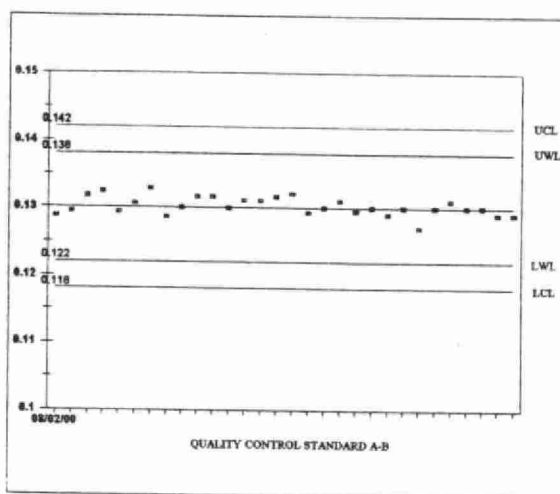
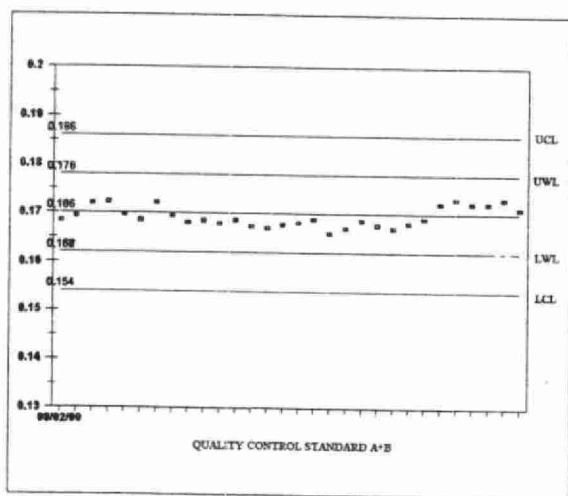
REFERENCE MATERIAL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
KCN:	30	0.10	0.0984	-0.0016	0.0082
FeCN:	30	0.10	0.0932	-0.0068	0.0057

DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
68	0 - 0.020	0.0012	36.2
5	0.021 - 0.040	0.0004	1.8
7	0.041 - 0.100	0.0005	0.8
2	0.101 - 0.200	0.0011	N.A.
82	Overall	0.0011	

CYANIDE TOTAL (mg/L as CN)
QUALITY CONTROL DATA FROM 08/02/00 TO 22/12/00
E3015



FLUORIDE

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	before '74
Method Reference No	E3369	Reporting Unit	mg/L as F
LIMS Product Code	F3369	Supervisor	P. WILSON
Sample Type/Matrix	Effluent, Industrial Waste, Process Water, Drinking Water, Ground Water, Leachate, Surface Water		

SAMPLING:

Quantity Required	50 mL
Container	Plastic

ANALYTICAL PROCEDURE:

Using an automated flow system the sample is distilled in the presence of sulphuric acid at 160°C; the distillate is then reacted (in an acetic acid-acetate buffer media) with Alizarin Fluorine Blue and lanthanum nitrate to form a ternary Alizarin Blue-lanthanide-fluoride complex.
Approximate absorbance: 0.8 at the full scale level.

INSTRUMENTATION:

Modular continuous flow colourimetric system plus a distillation module. Colourimetric measurement is through a 5.0 cm. light path at 630 nm. Data capture, reduction, and processing via a multi-stage microcomputer system.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.01	Current T value: 0.05
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CALIBRATION:

BL plus 6 standards

CONTROLS:

Calibration	LTB plus 3 standards, e.g. QCA
Drift	BL every 10 samples; standard every 20 samples

NOTES:

The HP data capture/processing was replaced by Labtronics in November 1999.

Feb 1998. The Nitrite test was discontinued at E3369 and made available at E3364 and E3366. The LIMS product code changed from FNOT3369 to F3369.

FLUORIDE (E3369)

QUALITY CONTROL DATA FROM 17/01/00 TO 29/12/900

Laboratory Unit: Colourimetry

Full Scale: to 2.0 mg/L as F

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	41	1.6	1.612	0.012	0.0222
B:	41	0.8	0.796	-0.004	0.0248
C:	41	0.16	0.158	-0.002	0.0124
A+B:		2.4	2.408	0.008	0.0320
A-B:		0.8	0.816	0.016	0.0345
B+C:		0.96	0.953	-0.007	0.0332
B-C:		0.64	0.638	-0.002	0.0208

s.d.(AB) S(between runs):0.0229

Sw(within run): 0.0242

S/Sw: 0.9

s.d.(BC) S(between runs):0.0207

Sw(within run): 0.0156

S/Sw: 1.3

The calibration is accepted if the calibration control values obtained lie within the ranges:

2.302	-	2.498	for	A+B
0.726	-	0.874	for	A-B
0.91	-	1.01	for	B+C
0.59	-	0.69	for	B-C

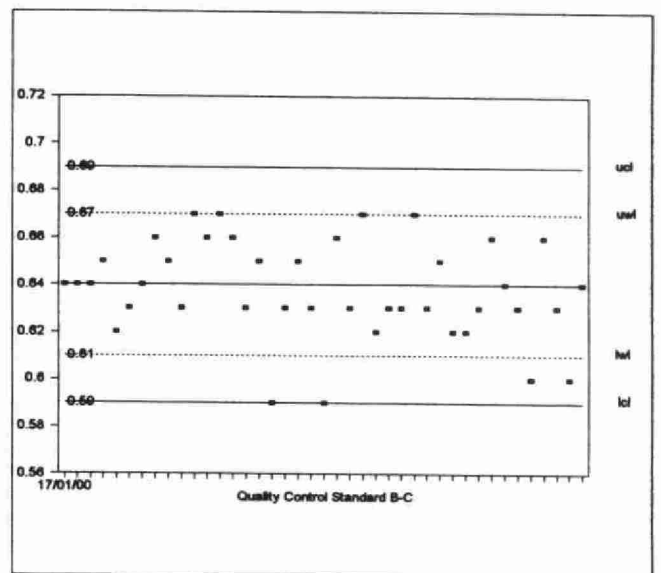
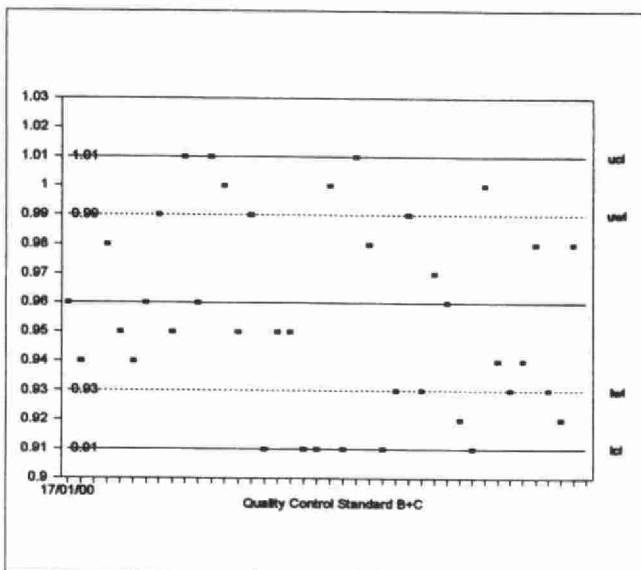
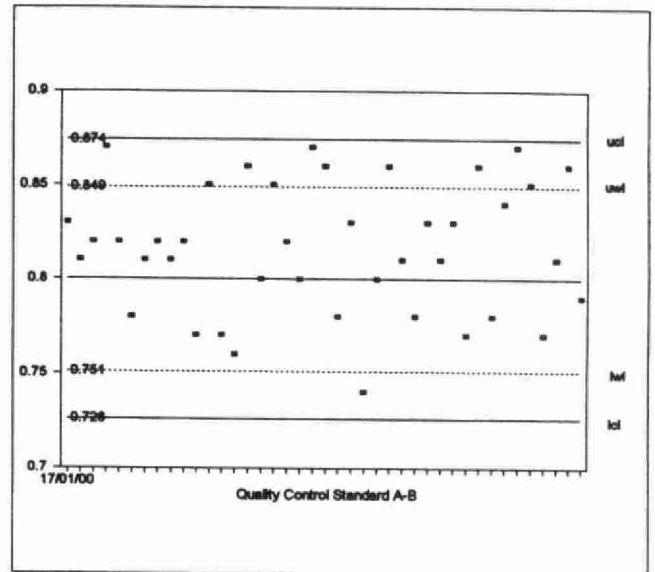
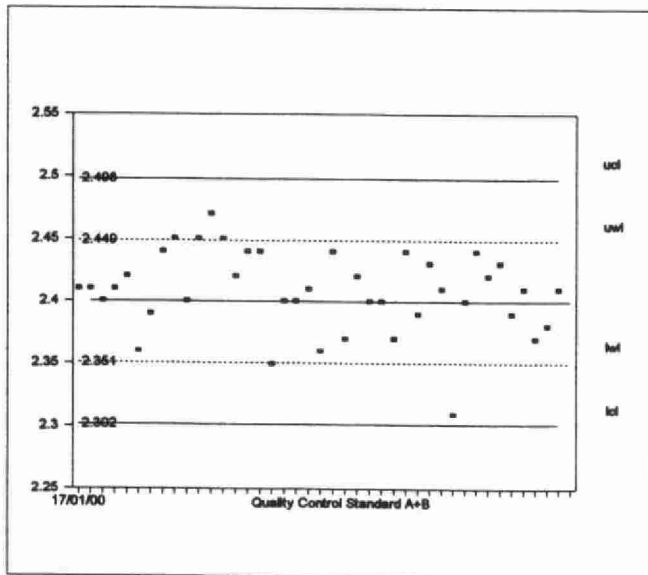
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
81	0.00 - 0.20	0.0133	13.6
7	0.21 - 0.40	0.0220	8.1
10	0.41 - 1.00	0.0129	2.0
17	1.01 - 2.00	0.0293	2.3
115	Overall	0.0172	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	42	0.0097	0.0252

Fluoride
 Quality Control Data From 17/01/00 To 29/12/00
 E3369



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 Canada

NITRATE

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/04/78
Method Reference No.	E3004	Units	$\mu\text{g}/\text{m}^3$ as NO_3
LIMS Product Code	ANION3004	Supervisor	P. Wilson
Sample Type/Matrix	Air; HiVol Glass Fibre, Quartz and Polyflon, Other Filters and Puff		

SAMPLING:

Quantity Required	3/4" or 1.9cm strip from 8"x10" filter
Container	50 mL polypropylene tube

SAMPLING PREPARATION:

A 3/4" strip is cut in pieces and deposited into a 50 mL polypropylene tube. 50 mL of Pure-Water is added to the tube. The tube is placed on a horizontal shaker for approximately 1 hour. The supernatant is then filtered into a 15 mL plastic tube and analysed.

ANALYTICAL PROCEDURE:

Nitrate separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of sodium bicarbonate and sodium carbonate and a conductivity detector. The concentration of nitrate (mg/L) is determined by the comparison of the analyte peak area count to that of a series of standards. The analyte result is corrected for the filter blank before the final calculation is made. The result is reported as $\mu\text{g}/\text{m}^3$ as NO_3 .

Chloride and sulphate are determined simultaneously.

INSTRUMENTATION:

Horizontal Shaker, ion chromatographic system plus a PC with ChromPerfect software and DT2804 card for automated sample injection, timing, and data processing.

REPORTING:

Maximum Significant Figures: 2	Current W value: $0.1 \mu\text{g}/\text{m}^3$	Current T value: $0.5 \mu\text{g}/\text{m}^3$
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CALIBRATION:

6 standards

CONTROLS:

Calibration	MB, IS(n), CS1, and CS2
Drift	Duplicate plus 2 standards approximately every 20 samples

NITRATE cont'd

NOTES:

To convert unit from mg/L to $\mu\text{g}/\text{m}^3$, the final concentration of NO_3 in mg/L is multiplied by the following formula:

$$\text{Result (mg/L)} \times 50\text{mL} \times (63/6.75) / \text{air volume} = \mu\text{g}/\text{m}^3$$

Where: 63 is the area of the filter exposed and 6.75 is the sample aliquot area in square inch.

NITRATE (E3004)

QUALITY CONTROL DATA FOR 2000

Analytical Range: to 28.61 $\mu\text{g}/\text{m}^3$ as NO_3

DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
69	0 - 2.86	0.049	6.6
14	2.89 - 7.15	0.118	2.8
2	7.18 - 14.31	N.A.	N.A.
0	14.33 - 24.90	N.A.	N.A.
85	Overall	0.072	

NITRILOTRIACETIC ACID

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	17/04/98
Method Reference No.	E3406	Reporting Unit	mg/L as NTA
LIMS Product Code	NTA3406	Supervisor	P. Wilson
Sample Type/Matrix	Drinking Water		

SAMPLING:

Quantity Required	50 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Nitrilotriacetic Acid is separated from other anions in the samples by automated suppressed gradient ion chromatography. A sodium hydroxide eluent is used with conductivity detection. The concentration of Nitrilotriacetic acid in mg/L as NTA is determined by comparison of the sample scan to a series of standard scans.

INSTRUMENTATION:

Basic modular continuous flow ion chromatographic system with gradient flow control module .

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.01	Current T value: 0.05
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CALIBRATION:

BL plus 7 standards

CONTROLS:

Calibration	LTBL plus 2 standards, e.g. QCA
Drift	1 standard every 10 samples

NITRILOTRIACETIC ACID (E3406)

QUALITY CONTROL DATA FROM 10/01/00 TO 15/12/00

Full Scale: to 1.00 mg/L as NTA

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	35	0.80	0.8025	0.0025	0.0136
B:	35	0.20	0.1972	-0.0028	0.0106
A+B:		1.00	0.9997	-0.0003	0.0200
A-B:		0.60	0.6053	0.0053	0.0139

s.d.(AB) S(between runs): 0.0122 Sw(within run): 0.0098 S/Sw: 1.2

The calibration is accepted if the calibration control values obtained lie within the ranges:

0.95 - 1.05 for A+B
0.56 - 0.64 for A-B

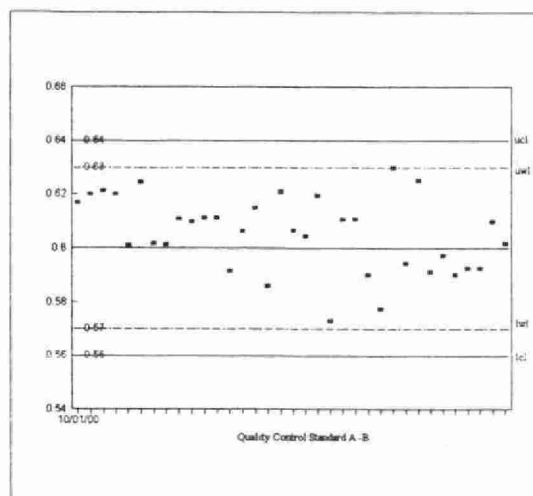
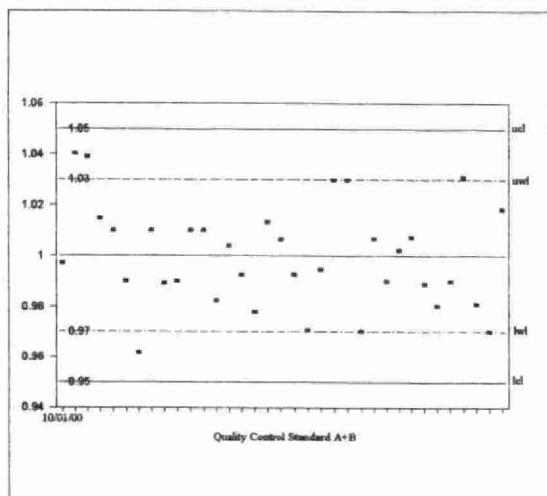
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
5	0.00 - 0.10	0.0066	7.8
10	0.10 - 0.20	0.0100	8.5
0	0.20 - 0.50	N.A.	N.A.
0	0.50 - 1.00	N.A.	N.A.
15	Overall		

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	35	0.0014	0.0059

NITRILOTRIACETIC ACID (E3406)
QUALITY CONTROL DATA FROM 10/01/00 TO 15/12/00
E3406



NITROGEN, AMMONIA PLUS AMMONIUM

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/04/78
Method Reference No.	E3364	Reporting Unit	mg/L as N
LIMS Product Code	DISNUT3364	Supervisor	P.Wilson
Sample Type/Matrix	Dried Sludge, Sediment, Soil, Vegetation, Drinking Water, Ground Water, Surface Water		

SAMPLING:

Quantity Required	10 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Ammonia plus ammonium ions are determined on the supernatant of a settled sample via the formation of indophenol blue in a buffered system using nitroprusside as a catalyst. A reference stream, which differs from the colour formation stream by replacement of the catalyst with an equal flow of water, is employed to suppress sample matrix effects.

Approximate absorbance: 0.5 at the full scale level.

Nitrate plus nitrite, nitrite, and reactive orthophosphate are determined simultaneously.

INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: 2 of 37°C heating bath (7.7 mL delay). Colourimetric measurement is through a 1.5 cm. light path at 630 nm.

Data capture, reduction, and processing via a multi-stage microcomputer system.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.002	Current T value: 0.01
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CALIBRATION:

BL plus 7 standards

CONTROLS:

Calibration	LTBL plus 3 standards, e.g. QCA
Drift	BL, standard, and BL after every 10 samples

NOTES:

The HP data capture / processing system was replaced by Labtronics in August 1999.

NITROGEN, AMMONIA PLUS AMMONIUM (E3364)

QUALITY CONTROL DATA FROM 05/01/00 TO 28/12/00

Laboratory Unit: Colourimetry

Full Scale: to 2.00 mg/L as N

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	104	1.60	1.602	0.0017	0.0130
B:	104	0.800	0.794	-0.0059	0.0099
C:	104	0.160	0.159	-0.0012	0.0053
A+B:		2.40	2.396	-0.0039	0.0185
A-B:		0.800	0.808	0.0079	0.0139
B+C:		0.960	0.953	-0.0071	0.0113
B-C:		0.640	0.635	-0.0047	0.0111

s.d.(AB) S(between runs):0.0116

Sw(within run): 0.0102

S/Sw: 1.1

s.d.(BC) S(between runs):0.0079

Sw(within run): 0.0077

S/Sw: 1.0

The calibration is accepted if the calibration control values obtained lie within the ranges:

2.353	-	2.447	for	A+B
0.765	-	0.835	for	A-B
0.931	-	0.989	for	B+C
0.618	-	0.662	for	B-C

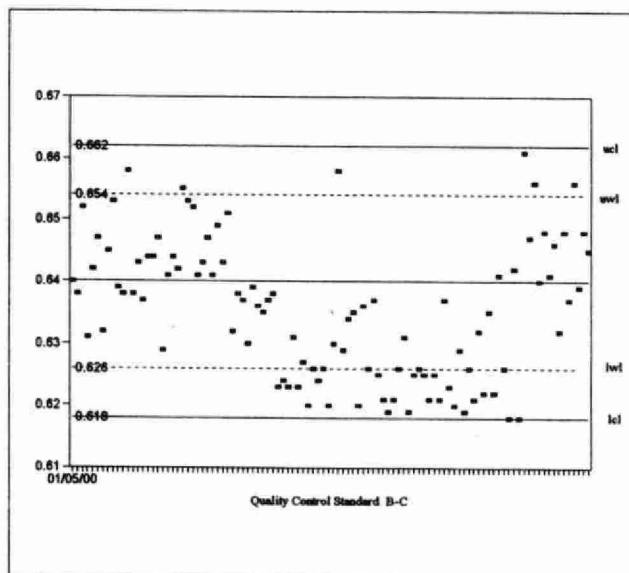
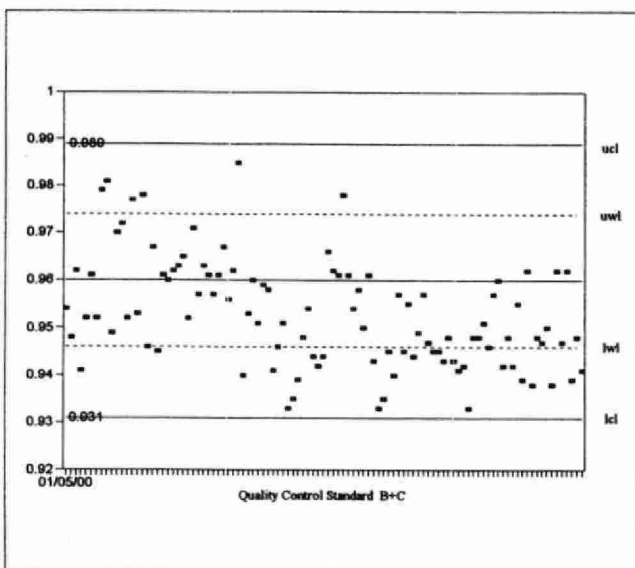
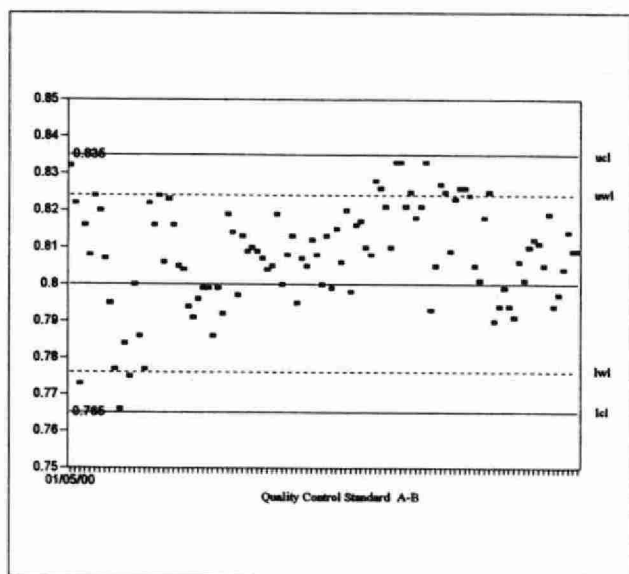
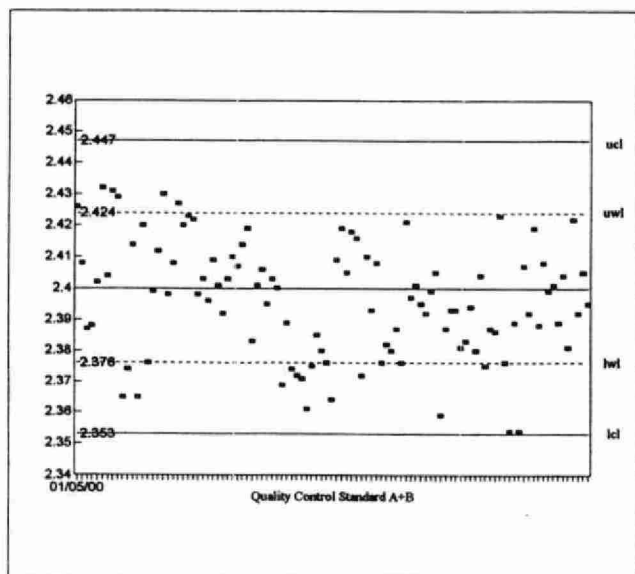
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
277	0.000 - 0.200	0.003	9.6
5	0.201 - 0.400	0.0163	5.5
12	0.401 - 1.00	0.0307	0.6
1	1.01 - 2.00	0.0495	N.A.
295	Overall	0.0047	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	104	0.0025	0.0050

NITROGEN, AMMONIA PLUS AMMONIUM (mg/L as N)
QUALITY CONTROL DATA FROM 05/01/00 TO 28/12/00
E3364



NITROGEN, AMMONIA PLUS AMMONIUM

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/04/77
Method Reference No.	E3366	Reporting Unit	mg/L as N
LIMS Product Code	DISNUT3366	Supervisor	P.Wilson
Sample Type/Matrix	Sludge, Effluent, Raw Sewage, Industrial Waste, Process Water, Leachate, Drinking Water, Ground Water, Surface Water.		

SAMPLING:

Quantity Required	10 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Ammonia plus ammonium ions are determined on the supernatant of a settled sample via the formation of indophenol blue in a buffered system using nitroprusside as a catalyst.

Approximate absorbance: 0.7 at the full scale level.

Reactive orthophosphate, nitrogen-nitrite and nitrogen-nitrate plus nitrite are determined simultaneously.

INSTRUMENTATION:

Basic automated modular continuous flow system plus one 38°C heating bath (7.7 mL delay). Colourimetric measurement is through a 1.5 cm. light path at 630 nm. Data capture, reduction, and processing via a multi-stage microcomputer system.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.05	Current T value: 0.25
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CALIBRATION:

BL plus 7 standards

CONTROLS:

Calibration	LTBL plus 3 standards, e.g. QCA
Drift	BL, standard and BL every 10 samples

NOTES:

The HP capture / processing system was replaced by Labtronics in October 1999.

NITROGEN, AMMONIA PLUS AMMONIUM (E3366)

QUALITY CONTROL DATA FROM 06/01/00 TO 21/12/00

Laboratory Unit: Colourimetry

Full Scale: to 50.0 mg/L as N

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	78	40.0	39.854	-0.146	0.2532
B:	78	20.0	19.986	-0.014	0.1555
C:	78	4.00	3.982	-0.018	0.0927
A+B:		60.0	59.840	-0.160	0.3457
A-B:		20.0	19.869	-0.131	0.2389
B+C:		24.0	23.968	-0.032	0.2038
B-C:		16.0	16.004	0.004	0.1550

s.d.(AB) S(between runs):0.21
s.d.(BC) S(between runs):0.13

Sw(within run): 0.17 S/Sw: 1.1
Sw(within run): 0.11 S/Sw: 1.2

The calibration is **accepted** if the calibration control values obtained lie within the ranges:

58.76	-	61.24	for	A+B
19.07	-	20.93	for	A-B
23.34	-	24.66	for	B+C
15.50	-	16.50	for	B-C

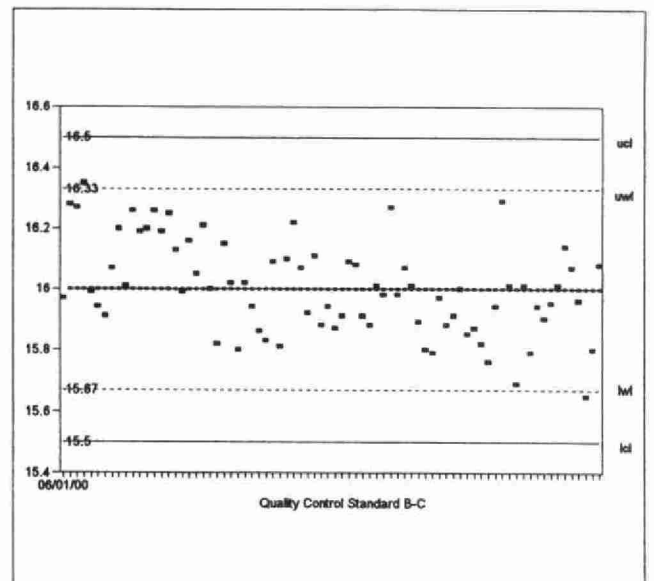
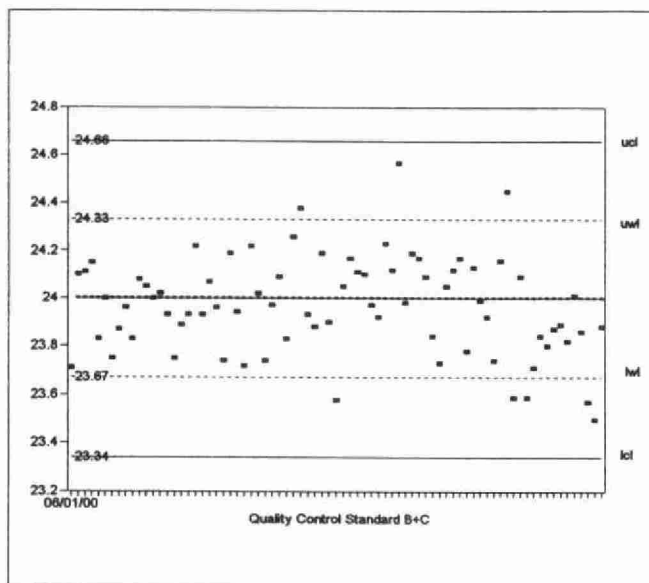
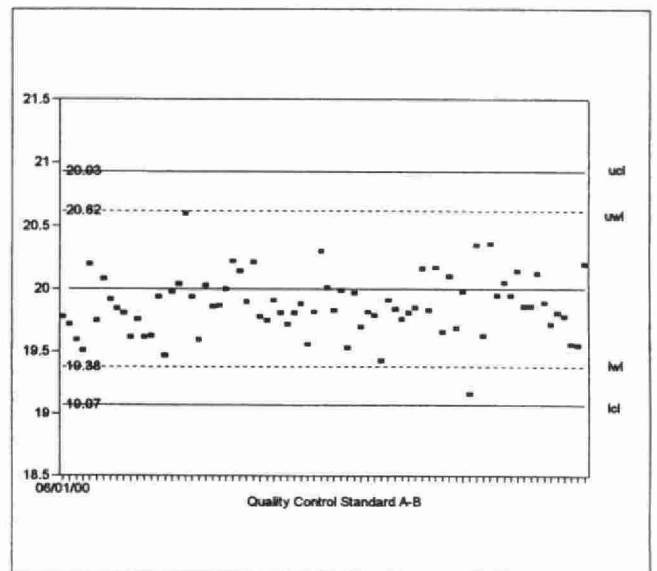
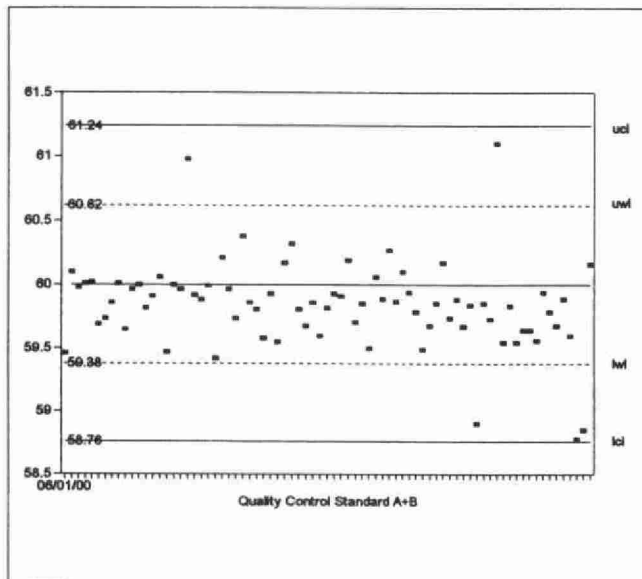
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
176	0 - 5.00	0.0395	6.3
17	5.1 - 10.0	0.0649	0.9
16	10.1 - 25.0	0.1087	1.2
3	25.1 - 50.0	0.3723	2.4
212	Overall	0.0670	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	78	0.1542	0.1247

NITROGEN, AMMONIA PLUS AMMONIUM (mg/L as N)
QUALITY CONTROL DATA FROM 06/01/00 TO 21/12/00
E3366



NITROGEN, NITRATE PLUS NITRITE

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/04/78
Method Reference No.	E3364	Reporting Unit	mg/L as N
LIMS Product Code	DISNUT3364	Supervisor	P. Wilson
Sample Type/Matrix	Dried Sludge, Sediment, Soil, Vegetation, Drinking Water, Ground Water, Surface Water		

SAMPLING:

Quantity Required	10 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Nitrate plus nitrite is determined on the supernatant of a settled sample. Nitrate is reduced to nitrite in alkaline media at 37°C, by hydrazine sulphate with copper as a catalyst. Colourimetry is based on the formation of an azo dye by nitrite, sulphanilamide, and N(1-naphthyl) ethylenediamine dihydrochloride. To control metal ion interference, samples are passed through an ion-exchange column prior to the reduction step. Approximate absorbance: 0.6 at the full scale level.

Ammonia plus ammonium, nitrite, and reactive orthophosphate are determined simultaneously.

INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: 37°C heating bath (7.7 mL delay), ion exchange column. Colourimetric measurement is through a 1.5 cm. light path at 520 nm. Data capture, reduction, and processing via a multi-stage microcomputer system.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.005	Current T value: 0.025
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CALIBRATION:

BL plus 7 standards

CONTROLS:

Calibration	LTBL plus 3 standards, e.g. QCA
Drift	BL, standard and BL every 10 samples
Interference	Nitrate standard spiked with calcium (150 mg/L) and magnesium (50 mg/L) confirms effective interference suppression.
Recovery	Individual nitrate and nitrite standards of equal N concentration show effectiveness of reduction step.

NOTES:

The HP data capture / processing system was replaced by Labtronics in August 1999.

NITROGEN, NITRATE PLUS NITRITE (E3364)

QUALITY CONTROL DATA FROM 05/01/00 TO 28/12/00

Laboratory Unit: Colourimetry

Full Scale: to 5.00 mg/L as N

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	MeanBias	Standard Deviation (1)
A:	104	4.00	4.006	0.006	0.0385
B:	104	2.00	2.002	0.002	0.0234
C:	104	0.40	0.396	-0.004	0.0096
A+B:		6.00	6.008	0.008	0.0484
A-B:		2.00	2.005	0.005	0.0414
B+C:		2.40	2.397	-0.003	0.0289
B-C:		1.60	1.606	0.006	0.0207

s.d.(AB) S(between runs):0.0311
s.d.(BC) S(between runs):0.0179

Sw(within run): 0.0285 S/Sw: 1.1
Sw(within run): 0.0152 S/Sw: 1.2

The calibration is accepted if the calibration control values obtained lie within the ranges:

5.868 - 6.132 for A+B
1.901 - 2.099 for A-B
2.328 - 2.472 for B+C
1.546 - 1.654 for B-C

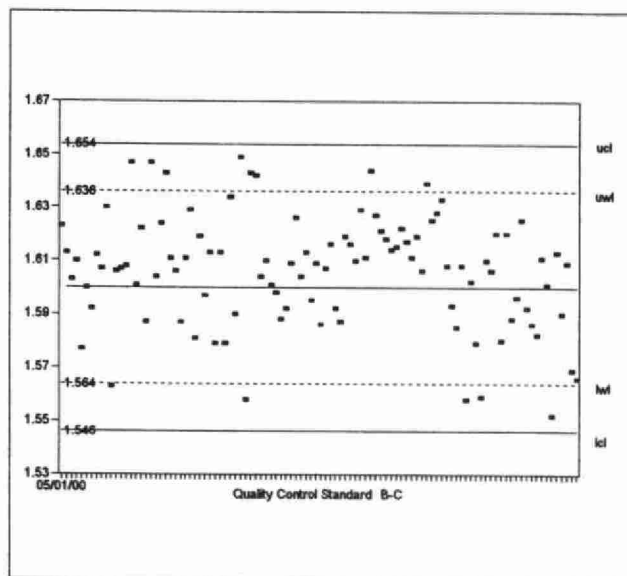
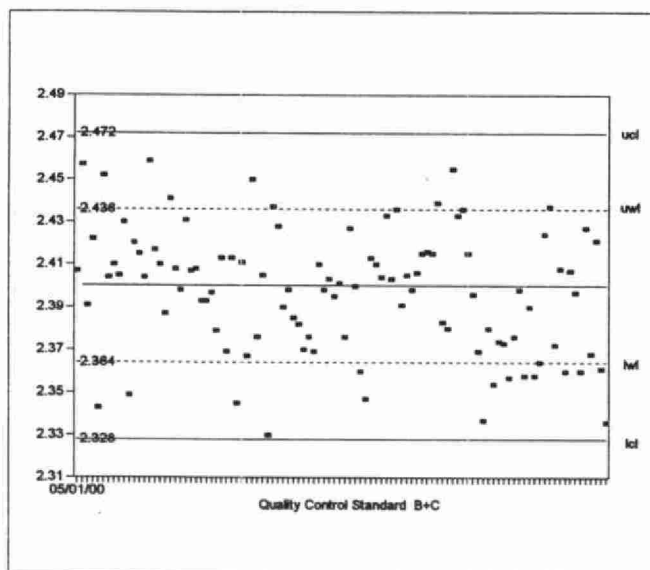
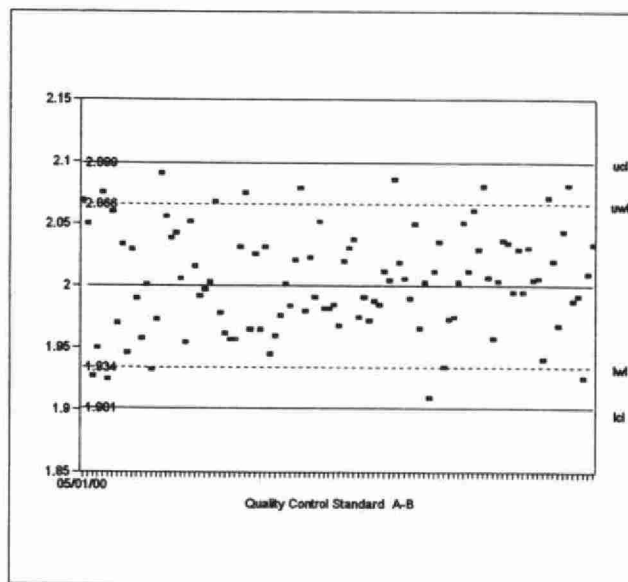
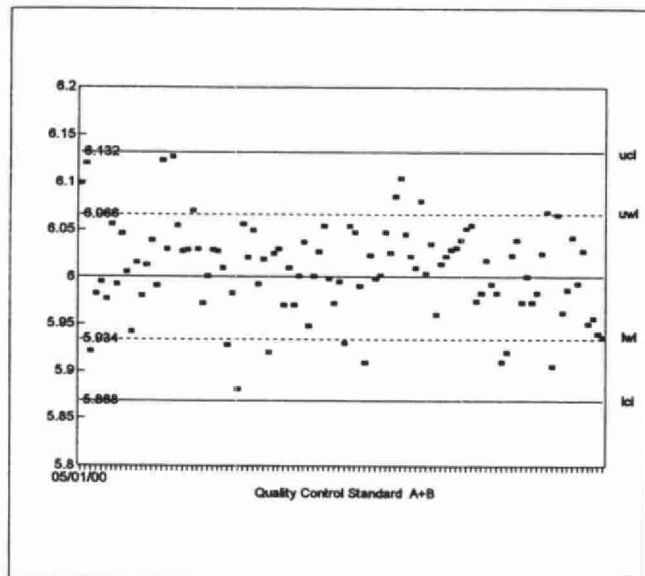
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
189	0 - 0.500	0.0073	4.2
32	0.501 - 1.00	0.0070	1.0
34	1.01 - 2.50	0.0186	1.1
29	2.51 - 5.00	0.0269	0.8
284	Overall	0.0125	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	104	0.0016	0.0073

NITROGEN, NITRATE PLUS NITRITE (mg/L as N)
QUALITY CONTROL DATA FROM 05/01/00 TO 28/12/00
E3364



NITROGEN, NITRATE PLUS NITRITE

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/04/78
Method Reference No.	E3366	Reporting Unit	mg/L as N
LIMS Product Code	DISNUT3366	Supervisor	P.Wilson
Sample Type/Matrix	Sludge, Effluent, Raw Sewage, Industrial Waste, Process Water, Leachate, Drinking Water, Ground Water, Surface Water.		

SAMPLING:

Quantity Required	10 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Nitrate plus nitrite is determined on the supernatant of a settled sample. Nitrate is reduced to nitrite in alkaline media at 38°C, by hydrazine sulphate with copper as a catalyst. Colourimetry is based on the formation of an azo dye by nitrite, sulphanilamide, and N(1-naphthyl) ethylenediamine dihydrochloride. To control metal ion interference, samples are passed through an ion-exchange column prior to the reduction step. Approximate absorbance: 0.7 at the full scale level.

Ammonia plus ammonium, nitrite, and reactive phosphate are determined simultaneously.

INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: 38°C heating bath (7.7 mL delay). Colourimetric measurement is through a 1.5 cm. light path at 520 nm. Two analytical ranges are obtained from the output of the colourimeter. Data capture, reduction, and processing via a multi - stage microcomputer system.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.05	Current T value: 0.25
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CALIBRATION:

BL plus 7 standards

CONTROLS:

Calibration	LTBL plus 3 standards, e.g. QCA
Drift	BL ,standard and BL every 10 samples
Interference	Nitrate standard spiked with calcium (150 mg/L) and magnesium (50mg/L) confirms effective interference suppression.
Recovery	Individual nitrate and nitrite standards of equal N concentration show effectiveness of reduction step.

NOTES:

The HP capture / processing system was replaced by Labtronics in October 1999.

NITROGEN, NITRATE PLUS NITRITE (E3366)

QUALITY CONTROL DATA FROM 06/01/00 TO 21/12/00

Laboratory Unit: Colourimetry

Full Scale: to 50.0 mg/L as N

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	78	40.0	39.612	-0.388	0.4226
B:	78	20.0	19.971	-0.029	0.2316
C:	78	4.0	3.917	0.083	0.1701
A+B:		60.0	59.583	-0.417	0.5445
A-B:		20.0	19.642	-0.358	0.4099
B+C:		24.0	23.888	-0.112	0.2958
B-C:		16.0	16.053	0.053	0.2787

s.d.(AB) S(between runs):0.34

Sw(within run): 0.29

S/Sw: 1.02

s.d.(BC) S(between runs):0.20

Sw(within run): 0.20

S/Sw: 1.1

The calibration is accepted if the calibration control values obtained lie within the ranges:

58.66	-	61.34	for	A+B
18.99	-	21.01	for	A-B
23.28	-	24.72	for	B+C
15.46	-	16.54	for	B-C

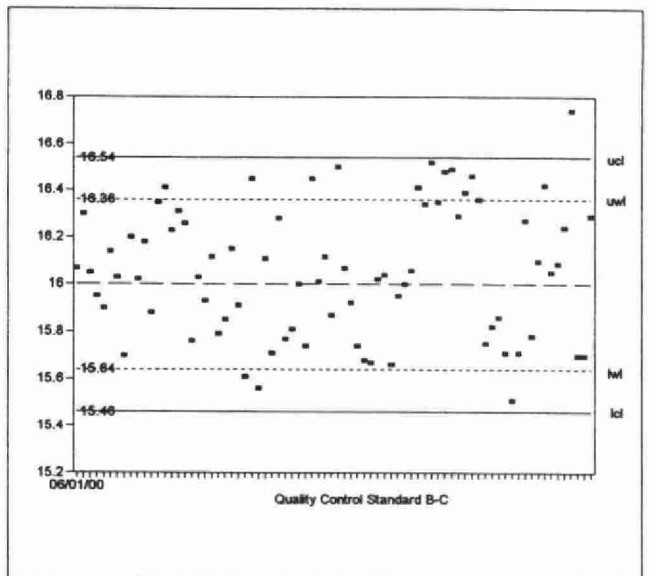
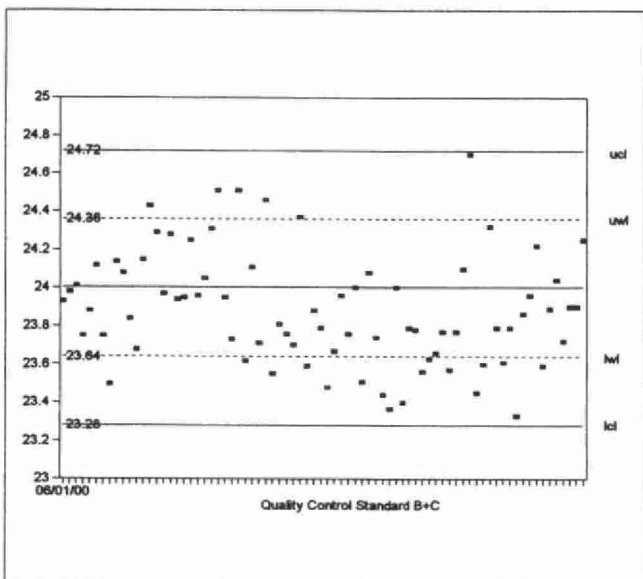
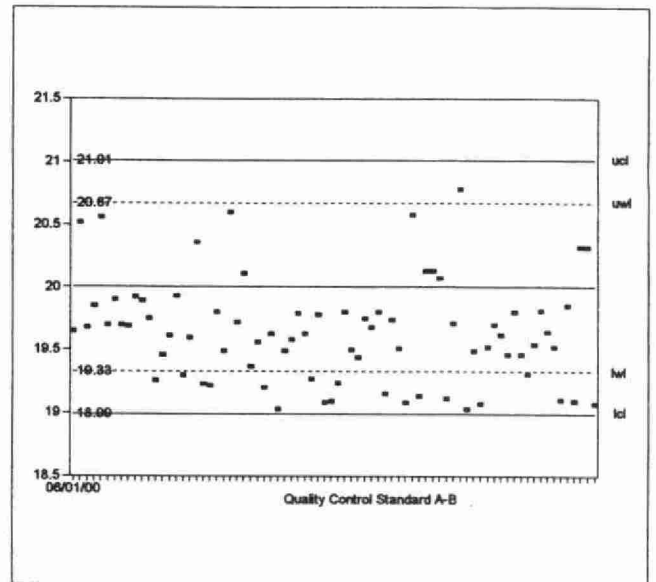
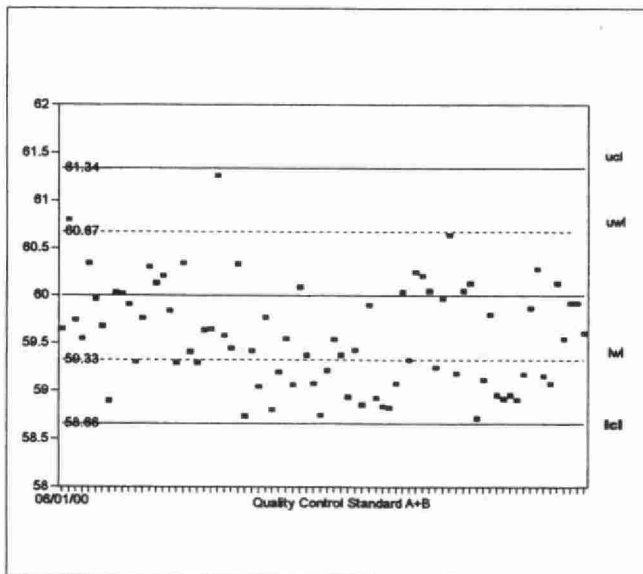
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
156	0.000 - 5.00	0.0659	9.0
24	5.01 - 10.0	0.0564	0.9
29	10.1 - 25.0	0.1709	1.2
6	25.1 - 50.0	0.1297	0.4
215	Overall	0.0890	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	78	-0.0086	0.0678

NITROGEN, NITRATE PLUS NITRITE (mg/L as N)
QUALITY CONTROL DATA FROM 06/01/00 TO 21/12/00
E3366



NITROGEN, NITRITE

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/04/78
Method Reference No.	E3364	Reporting Unit	mg/L as N
LIMS Product Code	DISNUT3364	Supervisor	P. Wilson
Sample Type/Matrix	Dried Sludge, Sediment, Soil, Vegetation, Drinking Water, Ground Water, Surface Water		

SAMPLING:

Quantity Required	10 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Nitrite is determined on the supernatant of a settled sample by formation of an azo dye using sulphanilamide, and N(1-naphthyl) ethylenediamine dihydrochloride.

Approximate absorbance: 0.6 at the full scale level.

Ammonia plus ammonium, nitrate plus nitrite, and reactive orthophosphate are determined simultaneously.

INSTRUMENTATION:

Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 520 nm.

Data capture, reduction, and processing via a multi-stage microcomputer system.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.001	Current T value: 0.005
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CALIBRATION:

BL plus 7 standards

CONTROLS:

Calibration	LTBL plus 3 standards, e.g. QCA
Drift	BL, standard and BL after every 10 samples
Interference	Nitrate standard spiked with calcium (150 mg/L) and magnesium (50 mg/L) confirms effective interference suppression.
Recovery	Individual nitrate and nitrite standards of equal N concentration show effectiveness of reduction step.

NOTES:

The HP data capture / processing system was replaced by Labtronics in August 1999.

NITROGEN, NITRITE (E3364)

QUALITY CONTROL DATA FROM 05/01/00 TO 28/12/00

Laboratory Unit: Colourimetry

Full Scale: to 0.200 mg/L as N

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	104	0.160	0.1597	-0.0003	0.0014
B:	104	0.080	0.0794	-0.0006	0.0012
C:	104	0.016	0.0156	-0.0004	0.0009
A+B:		0.240	0.2277	-0.0123	0.0515
A-B:		0.080	0.0764	-0.0036	0.0173
B+C:		0.096	0.0904	-0.0056	0.0205
B-C:		0.064	0.0608	-0.0032	0.0138

s.d.(AB) S(between runs):0.0013

Sw(within run): 0.0011

S/Sw: 1.18

s.d.(BC) S(between runs):0.0010

Sw(within run): 0.0009

S/Sw: 1.11

The calibration is accepted if the calibration control values obtained lie within the ranges:

0.234	-	0.246	for	A+B
0.076	-	0.084	for	A-B
0.092	-	0.100	for	B+C
0.061	-	0.067	for	B-C

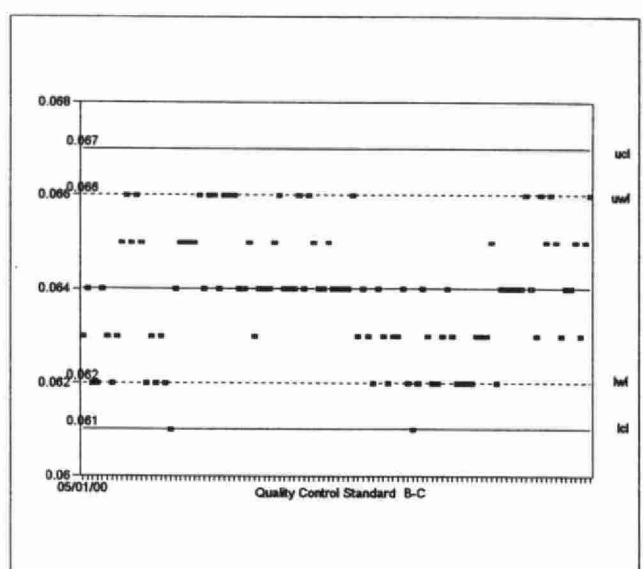
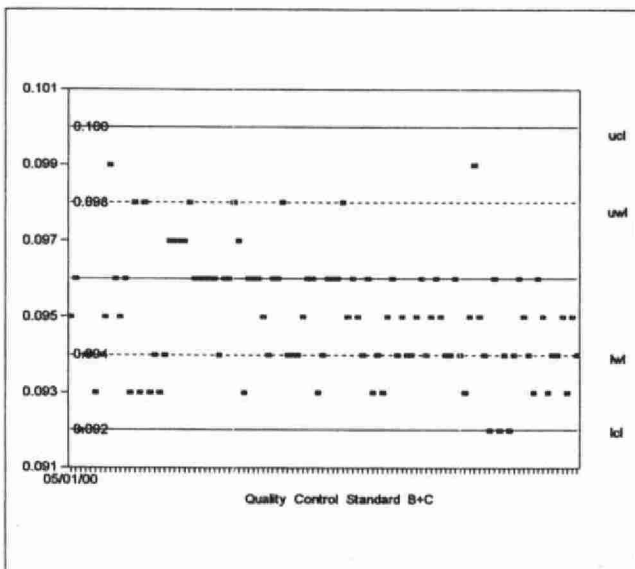
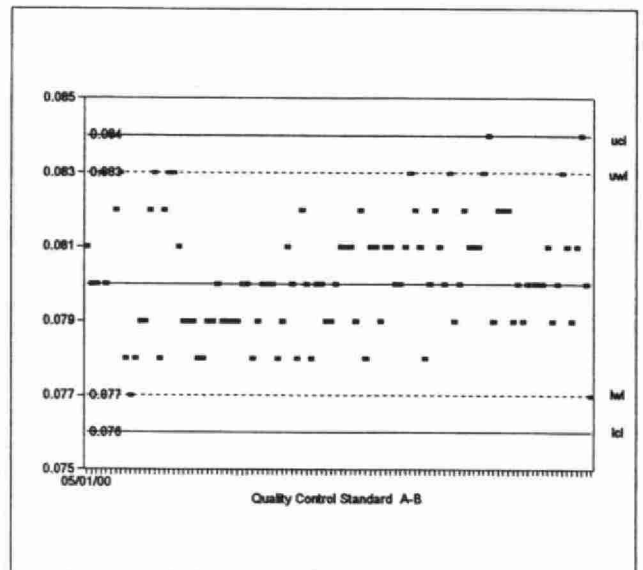
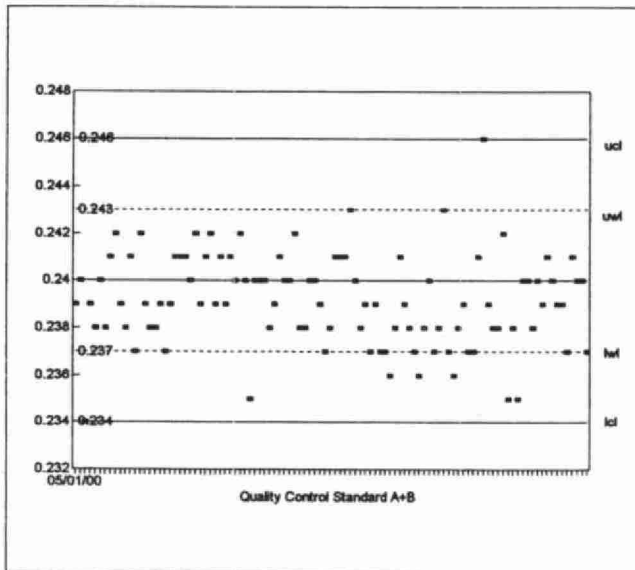
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
256	0 - 0.020	0.0010	24.7
15	0.021 - 0.040	0.0011	3.7
12	0.041 - 0.100	0.0020	3.2
9	0.101 - 0.200	0.0029	2.1
292	Overall	0.0012	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	104	0.00002	0.00111

NITROGEN, NITRITE (mg/L as N)
QUALITY CONTROL DATA FROM 05/01/00 TO 28/12/00
E3364



NITROGEN, NITRITE

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/04/78
Method Reference No	E3366	Reporting Unit	mg/L as N
LIMS Product Code	DISNUT3366	Supervisor	P.Wilson
Sample Type/Matrix	Sludge, Effluent, Raw Sewage, Industrial Waste, Process Water, Leachate, Drinking Water, Ground Water, Surface Water.		

SAMPLING:

Quantity Required	10 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Nitrite is determined on the supernatant of a settled sample by formation of an azo dye using sulphanilamide, and N(1-naphthyl) ethylenediamine dihydrochloride.

Approximate absorbance: 0.3 at the full scale level.

Ammonia plus ammonium, nitrate plus nitrite, and reactive orthophosphate are determined simultaneously.

INSTRUMENTATION:

Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 520 nm. Data capture, reduction, and processing via a multi-stage microcomputer system.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.005	Current T value: 0.025
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CALIBRATION:

BL plus 7 standards

CONTROLS:

Calibration	LTBL plus 3 standards, e.g. QCA
Drift	BL ,standard and BL every 10 samples
Interference	Nitrate standard spiked with calcium (150 mg/L) and magnesium (50 mg/L) confirms effective interference suppression.
Recovery	Individual nitrate and nitrite standards of equal N concentration show effectiveness of reduction step.

NOTES:

The HP capture / processing system was replaced by Labtronics in October 1999.

NITROGEN, NITRITE (E3366)

QUALITY CONTROL DATA FROM 06/01/00 TO 21/12/00

Laboratory Unit: Colourimetry

Full Scale: to 2.00 mg/L as N

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	78	1.60	1.6012	0.001	0.0174
B:	78	0.80	0.7917	-0.008	0.0075
C:	78	0.160	0.1613	0.001	0.0080
A+B:		2.40	2.3929	-0.007	0.0220
A-B:		0.80	0.8096	0.010	0.0152
B+C:		0.960	0.9529	-0.007	0.0096
B-C:		0.640	0.6304	-0.010	0.0119

s.d.(AB) S(between runs):0.0135
s.d.(BC) S(between runs):0.0071

Sw(within run): 0.0107 S/Sw: 1.3
Sw(within run): 0.0064 S/Sw: 1.1

The calibration is accepted if the calibration control values obtained lie within the ranges:

2.352 - 2.448 for A+B
0.764 - 0.836 for A-B
0.936 - 0.984 for B+C
0.622 - 0.658 for B-C

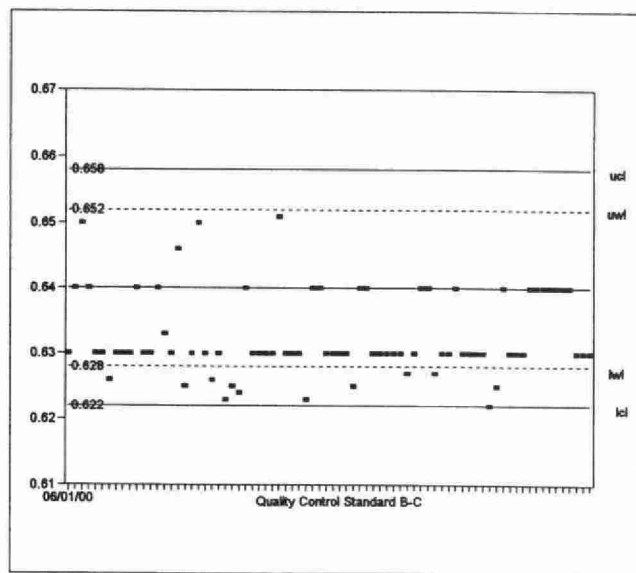
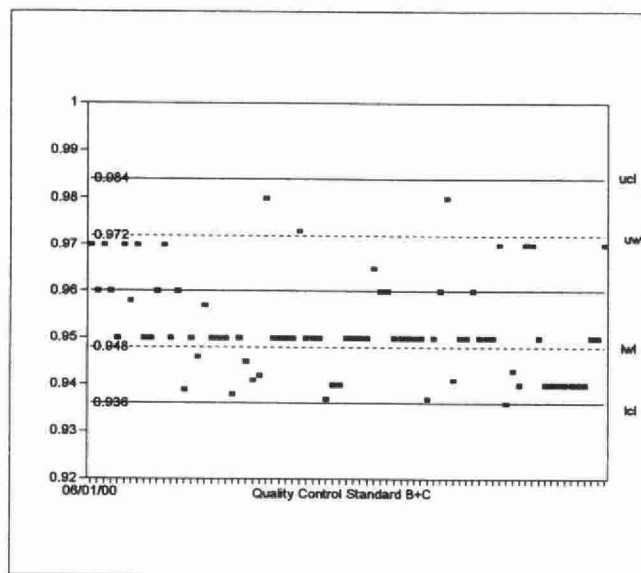
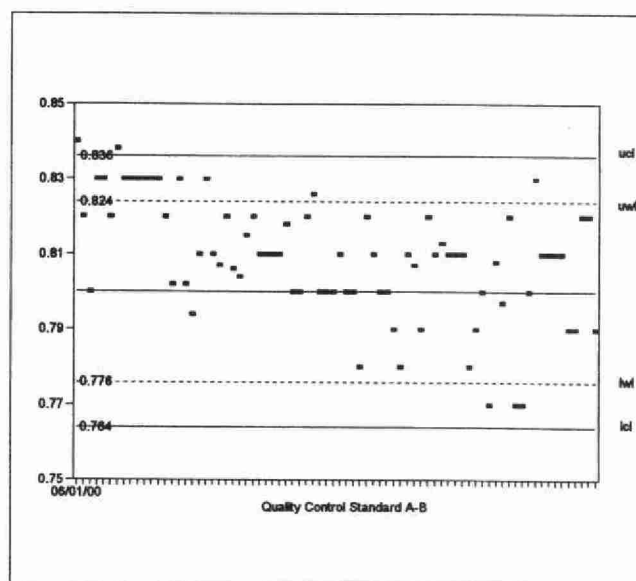
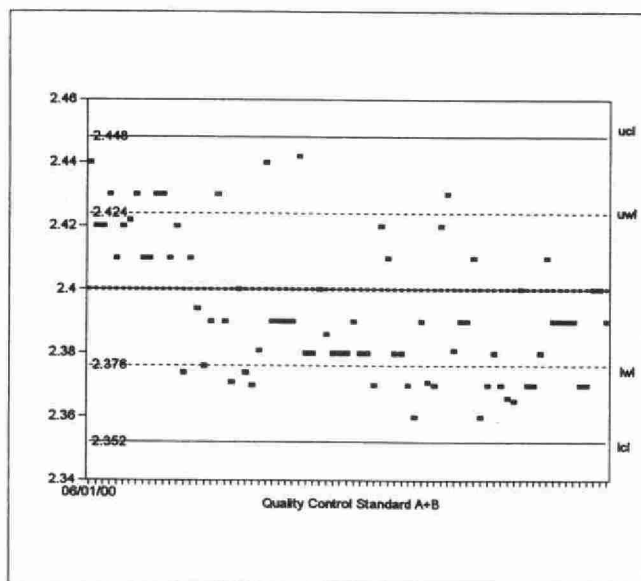
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
195	0.000 - 0.200	0.0067	27.8
9	0.201 - 0.400	0.0075	2.4
15	0.401 - 1.00	0.0063	0.9
3	1.01 - 2.00	0.0135	1.0
222	Overall	0.0069	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	78	-0.00808	0.0043

NITROGEN. NITRITE (mg/L as N)
QUALITY CONTROL DATA FROM 06/01/00 TO 21/12/00
E3366



NITROGEN, TOTAL KJELDAHL

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	Mar '89
Method Reference No.	E3116	Reporting Unit	mg/g as N
LIMS Product Code	TNP3116	Supervisor	P. Wilson
Sample Type/Matrix	Soil, Sediment, Dried Sludge		

SAMPLING:

Quantity Required	0.08 to 0.4 g
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Nitrogen compounds are converted to simple inorganic forms by dissolution of the samples in hot sulphuric acid and potassium persulphate. Potassium persulphate is added later in the digestion to raise the boiling point and to provide a highly oxidizing environment to decompose the more resistant organic matter. The digestate is filtered and the filtrate is analyzed using an automated colourimetric system.

INSTRUMENTATION:

Hot plate .

Basic automated modular continuous flow system : 37.5°C bath. Colourimetric measurement is through a 5 cm. light path at 630 nm.

Data capture, and processing via a microcomputer system.

REPORTING:

Maximum Significant Figures: 2 decimal places	Current W value: 0.1	Current T value: 0.5
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CALIBRATION:

3 High and 2 Low Calibration Standards

CONTROLS:

Calibration	In house composite B-Soil/sediment, plus QC Soils/Sediment (RS92)
Drift	4 BL's per run; high and low calibration standard at the end of the run
Recovery	1 digested BL plus 4 digested standards

NOTES:

System is calibrated with undigested standards.

NITROGEN, TOTAL KJELDAHL (E3116)

QUALITY CONTROL DATA FROM 12/01/00 TO 04/12/00

Full Scale: 10 mg/g as N

QUALITY CONTROL:

	n	Expected Concentration	Mean Concentration	Standard Deviation (1)
RS92 - In House Soil Composite	36	1.69	1.48	0.2215
RSM-2781 -Reference Standard Material	38	47.8	39.87	3.6788

The run is accepted if the control values obtained lie within the ranges:

1.39 - 1.99 for RS92
46.7 - 48.9 for RSM-2781

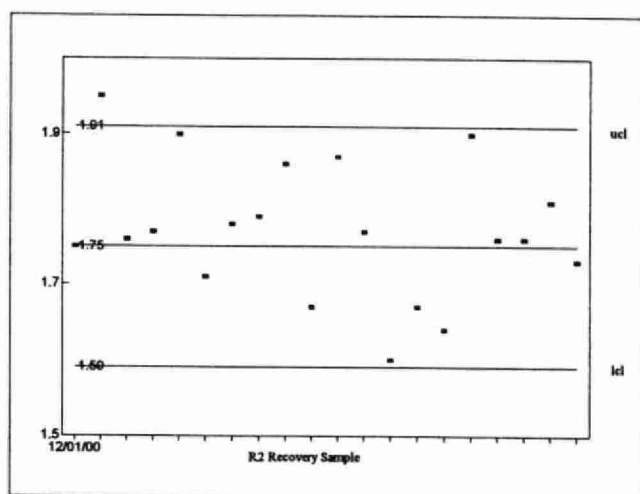
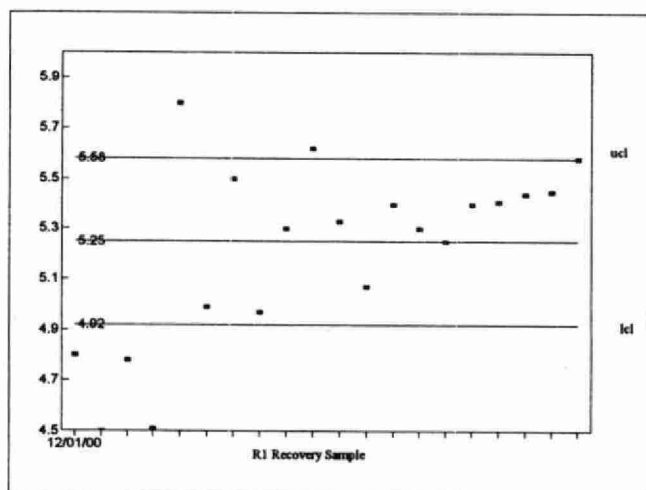
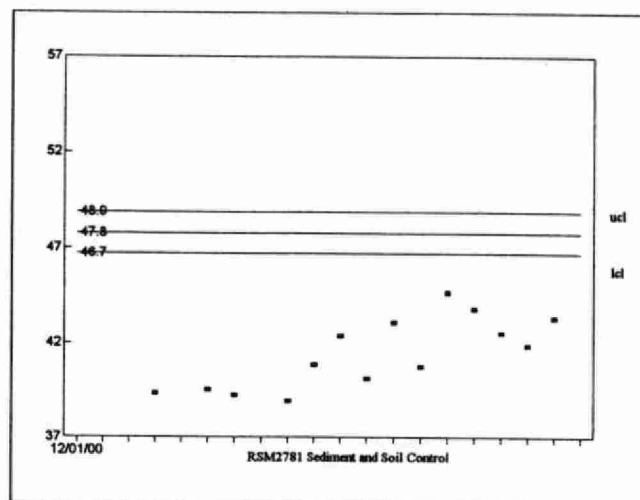
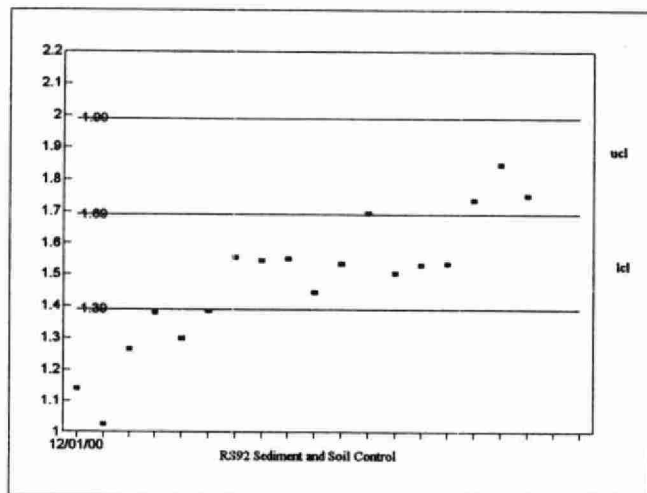
Recovery Standards

	n	Expected Concentration	Mean Concentration	Standard Deviation (1)
R1	20	5.25	5.22	0.3600
R2	20	1.75	1.77	0.0912

DUPLICATES: (Sediment/Soils)

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
14	0.0 - 2.0	0.1475	13.2
7	2.1 - 4.0	0.2610	8.4
16	4.1 - 10.0	0.2928	5.3
7	10.1 - 20.0	0.4512	3.6
44	Overall	0.2852	

NITROGEN, TOTAL KJELDAHL (mg/g as N)
QUALITY CONTROL DATA FROM 12/01/00 TO 04/12/00
E3116



NITROGEN, TOTAL KJELDAHL

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	Mar '89
Method Reference No.	E3118	Reporting Unit	mg/g as N
LIMS Product Code	TNP3118	Supervisor	P. Wilson
Sample Type/Matrix	Vegetation, Moss Bag		

SAMPLING:

Quantity Required	0.02 to 0.04 g
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Nitrogen compounds are converted to simple inorganic forms by dissolution of the samples in hot sulphuric acid and potassium persulphate. Potassium persulphate is added later in the digestion to raise the boiling point and to provide a highly oxidizing environment to decompose the more resistant organic matter. The digestate is analyzed using an automated colourimetric system.

INSTRUMENTATION:

Hot plate.

Basic automated modular continuous flow system : 37.5°C bath. Colourimetric measurement is through a 5 cm. light path at 630 nm.

Data capture, and processing via a microcomputer system.

REPORTING:

Maximum Significant Figures: 2 decimal places	Current W value: 0.20	Current T value: 1.00
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CALIBRATION:

3 High and 2 Low Calibration Standards

CONTROLS:

Calibration	In house composite A-VEG, plus QC VEG (Pine Needles)
Drift	4 BL's per run; high and low calibration standard at the end of the run
Recovery	1 digested BL plus 4 digested standards

NOTES:

System is calibrated with undigested standards.

There is not enough data to report QC for 2000.

NITROGEN, TOTAL KJELDAHL

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/04/79
Method Reference No.	E3367	Reporting Unit	mg/L as N
LIMS Product Code	TOTNUT3367	Supervisor	P.Wilson
Sample Type/Matrix	Precipitation, Drinking Water, Ground Water , Surface Water		

SAMPLING:

Quantity Required	50 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Samples are digested in a sulphuric acid-mercuric oxide-potassium sulphate media using three block digestors kept at 180°C, 210°C and 360°C. The pH of the digestate is adjusted in-line in two stages and then ammonia is determined by formation of indophenol blue in a buffered system using nitroprusside as a catalyst.

Approximate absorbance: 0.3 at the full scale level.

Total phosphorus is determined simultaneously.

INSTRUMENTATION:

Three block digesters

Basic automated modular continuous flow system plus 1 module: 38°C bath (7.7 mL delay). Colourimetric measurement is through a 5.0 cm. light path at 630 nm.

Data capture, reduction, and processing via a multi-stage microcomputer system

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.02	Current T value: 0.1
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CALIBRATION:

BL plus 7 undigested standards

CONTROLS:

Calibration	LTBL plus 3 undigested standards, e.g. QCA
Drift	BL, undigested standard , BL every 10 samples
Recovery	3 digested BL plus 3 digested standards in duplicate, e.g. R1

NOTE:

The HP capture / processing system was replaced by Labtronics in May 1999.

NITROGEN, TOTAL KJELDAHL (E3367)

QUALITY CONTROL DATA FROM 06/01/00 TO 29/12/00

Full Scale: to 2.00 mg/L as N

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	104	1.60	1.6007	0.0007	0.0140
B:	104	0.80	0.8013	0.0013	0.0104
C:	104	0.16	0.1598	-0.0002	0.0106
A+B:	104	2.40	2.3099	-0.0901	0.4647
A-B:	104	0.80	0.7682	-0.0318	0.1549
B+C:	104	0.96	0.9248	-0.0352	0.1866
B-C:	104	0.64	0.6169	0.0231	0.1244

s.d.(AB) S(between runs): 0.0127

Sw(within run): 0.0085

S/Sw: 1.5

s.d.(BC) S(between runs): 0.0112

Sw(within run): 0.0078

S/Sw: 1.4

The calibration is accepted if the calibration control values obtained lie within the ranges:

2.32	-	2.48	for	A+B
0.740	-	0.860	for	A-B
0.913	-	1.007	for	B+C
0.605	-	0.675	for	B-C

RECOVERIES:

Number of Data	Expected Concentration	Mean Concentration	Standard Deviation (1)
104	1.40	1.386	0.0387
104	0.840	0.828	0.0282
104	0.280	0.283	0.0163

DUPLICATES:

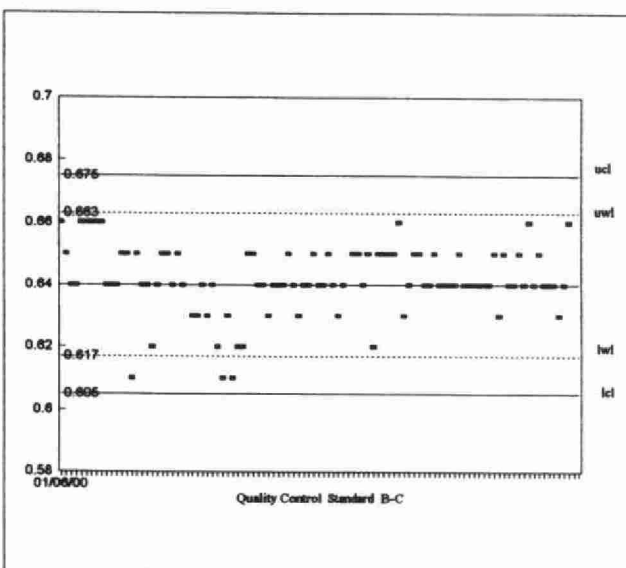
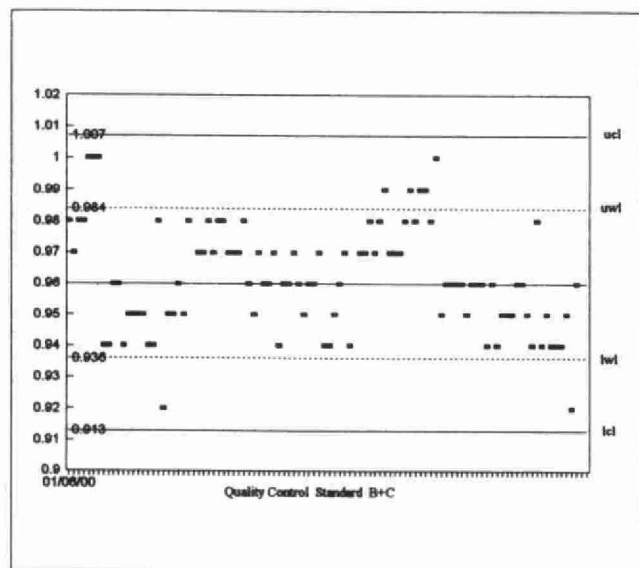
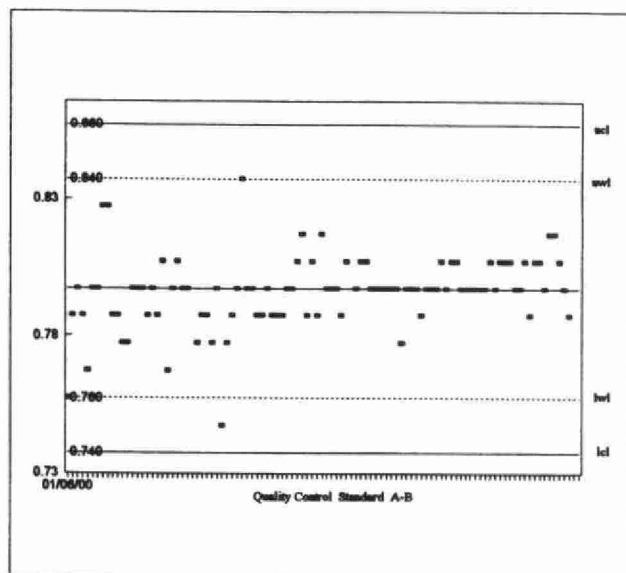
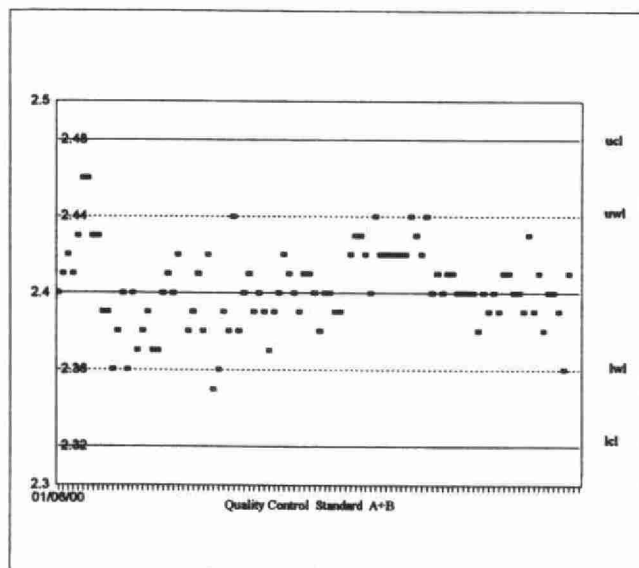
n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
67	0 - 0.20	0.0574	41.4
123	0.21 - 0.40	0.0429	14.6
88	0.41 - 1.00	0.0825	13.2
17	1.1 - 2.00	0.1341	10.9
295	Overall	0.0677	

NITROGEN, TOTAL KJELDAHL cont:

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	104	0.0094	0.0224
Digested Blank	104	0.0139	0.0128

NITROGEN, TOTAL KJELDAHL (mg/L as N)
QUALITY CONTROL DATA FROM 06/01/00 TO 29/12/00
E3367



NITROGEN, TOTAL KJELDAHL

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/04/79
Method Reference No	E3368	Reporting Unit	mg/L as N
LIMS Product Code	TOTNUT3368	Supervisor	P. Wilson
Sample Type/Matrix	Sludge, Raw Sewage, Industrial Waste, Drinking Water, Effluent, Ground Water, Process Water, Leachate, Surface Water.		

SAMPLING:

Quantity Required	50 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Samples are digested in a sulphuric acid-mercuric oxide-potassium sulphate media using three block digestors kept at 180°C, 210°C and 360°C. The pH of the digestate is adjusted in-line in two stages and then ammonia is determined by formation of indophenol blue in a buffered system using nitroprusside as a catalyst.

Approximate absorbance: 1.1 at the full scale level.

Total phosphorus is determined simultaneously.

INSTRUMENTATION:

Three block digesters

Basic automated modular continuous flow system plus 1 module: 38°C bath (7.7 mL delay). Colourimetric measurement is through a 1.5 cm. light path at 630 nm.

Data capture, reduction, and processing via a multi-stage microcomputer system

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.05	Current T value: 0.25
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CALIBRATION:

BL plus 7 undigested standards

CONTROLS:

Calibration	LTBL plus 3 undigested standards, e.g. QCA
Drift	BL every 10 samples; undigested standard every 20 samples
Recovery	3 digested BL plus 3 digested standards in duplicate, e.g. R1

NOTES:

System is calibrated with undigested standards.

The HP capture / processing system was replaced by Labtronics in April 1999.

NITROGEN, TOTAL KJELDAHL (E3368)

QUALITY CONTROL DATA FROM 18/01/00 TO 21/12/00

Full Scale: to 50.0 mg/L as N

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	51	40.0	40.066	0.066	0.2009
B:	51	20.0	20.108	0.108	0.1079
C:	51	4.0	3.975	-0.025	0.0713
A+B:		60.0	60.174	0.174	0.2486
A-B:		20.0	19.959	-0.041	0.2053
B+C:		24.0	24.083	0.083	0.1395
B-C:		16.0	16.132	0.132	0.1183

s.d.(AB) S(between runs): 0.161

Sw(within run): 0.145

S/Sw: 1.1

s.d.(BC) S(between runs): 0.091

Sw(within run): 0.084

S/Sw: 1.1

The calibration is accepted if the calibration control values obtained lie within the ranges:

59.27 - 60.73 for A+B
 19.45 - 20.55 for A-B
 23.58 - 24.42 for B+C
 15.68 - 16.32 for B-C

RECOVERIES:

Number of Data	Expected Concentration	Mean Concentration	Standard Deviation (1)
51	35	34.54	0.7071
51	21	20.77	0.4522
51	7	6.84	0.1753

DUPLICATES:

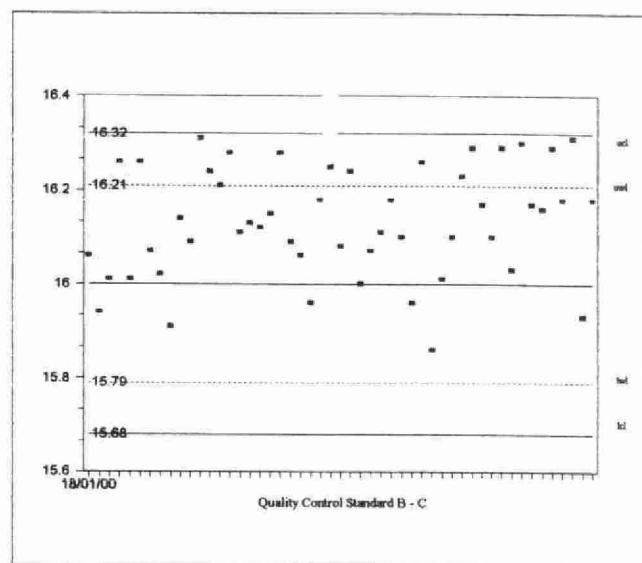
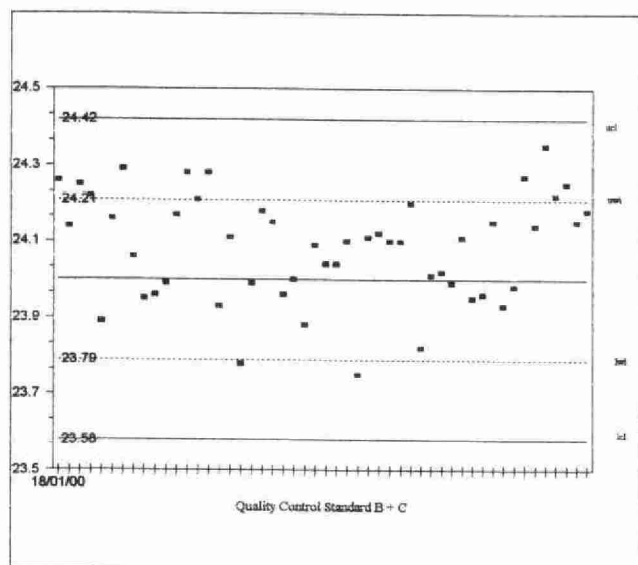
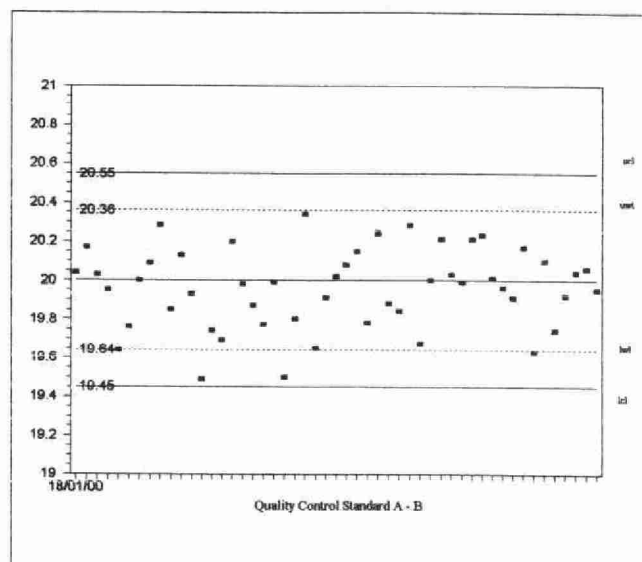
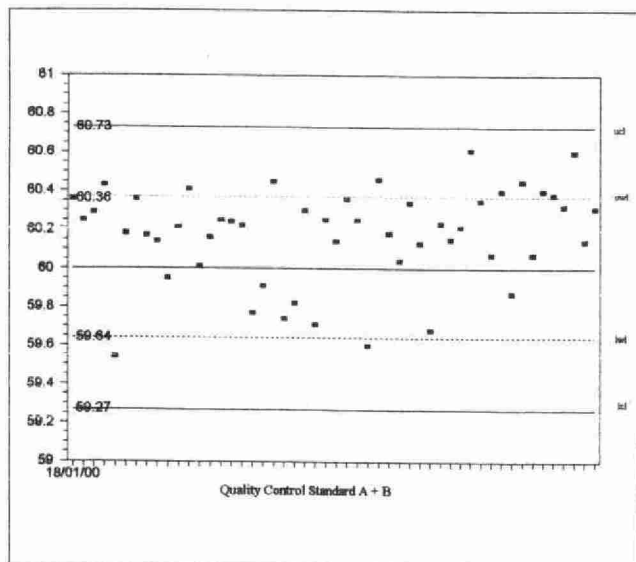
n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
116	0 - 5.0	0.1427	14.5
11	5.1 - 10.0	0.2998	4.0
14	10.1 - 25.0	0.3241	2.0
2	25.1 - 50.0	0.3276	1.2
143	Overall	0.1877	

NITROGEN, TOTAL KJELDAHL (E3368) cont'd

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	51	-0.005	0.0744
Digested Blank	51	-0.046	0.0733

NITROGEN, TOTAL KJELDAHL (mg/L as N)
QUALITY CONTROL DATA FROM 18/01/00 TO 21/12/00
E3368



OXYGEN DEMAND, BIOCHEMICAL

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	Before '61
Method Reference No.	E3182	Reporting Unit	mg/L as O
LIMS Product Code	BOD3182	Supervisor	P. Wilson
Sample Type/Matrix	Raw Sewage, Industrial Waste, Effluent, Drinking Water, Ground Water, Leachate, Surface Water		

SAMPLING:

Quantity Required:	400 mL
Container:	Glass or plastic

SAMPLE PREPARATION:

If necessary sample pH is adjusted to neutral and chlorine is removed by reaction with sodium sulphite.

ANALYTICAL PROCEDURE:

Oxygen depletion is measured as the difference in dissolved oxygen (DO) concentration. DO readings are taken prior to sample storage, and also at the end of storage in the dark at 20°C for five days (BOD5). If necessary, dilutions are made with aerated, nutrient-enriched water to obtain a 25-75% oxygen depletion. If the sample has undergone any of the sample preparation steps listed above or if the sample is an industrial waste, a sewage seed is added. For such samples, calculation of an appropriate seed correction is required.

INSTRUMENTATION:

- YSI Model 59 DO meter (Yellow Springs Instrument Company) with DO probe equipped with stirrer and fitted with a Teflon membrane of 0.5 mil thickness which is permeable to oxygen (1 mil = 0.001 inch).
- Titration equipment for Winkler analysis of dissolved oxygen.
- Incubator (19-21°C); BOD bottles (300 mL)

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.2	Current T value: 1
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CALIBRATION (DO):

The standard is air-saturated reversed osmosis deionized water. The DO content is read from a table (ORBISPHERE LABORATORIES - Pressure temperature dissolved oxygen table) after measuring its temperature and the barometric pressure in the laboratory.

OXYGEN DEMAND, BIOCHEMICAL cont'd

CONTROLS:

Calibration (DO)	2 QC solutions of Pure-DW water which have been partially stripped of DO by flushing with nitrogen. These "solutions", of different but unknown DO, are compared using the Oxygen meter and the Winkler titration procedure. The difference between the values for the two analytical methods is utilized as a slope control for the DO Analyzer.
Recovery (BOD5)*	3 Recovery standards prepared from a combination of Glucose and Glutamic Acid e.g. R1; the expected BOD5 is 67% of the oxygen requirement for complete oxidation.
Drift	Air saturated Pure-DW water after every 24 samples.
Blanks*	Pure-DW water and BOD dilution water

NOTES:

* These solutions are incubated for five days alongside samples.

OXYGEN DEMAND, BIOCHEMICAL (E3182)

QUALITY CONTROL DATA FROM 07/01/00 TO 27/12/00

Full Scale: to 9.0 mg/L as O at 20°C

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	96	0.00	-0.0126	-0.0126	0.0872
B:	96	0.00	-0.0013	-0.0013	0.0782

On any given day the calibration is accepted if the values obtained lie within the ranges:

-0.25 - 0.25

RECOVERIES:

Number of Data	Expected Depletion	Mean Depletion	Standard Deviation (1)
49	2.20	2.17	0.2008
49	4.34	4.30	0.2650
49	6.52	6.48	0.3608

DUPLICATES:

n Data Pairs	Sample Depletion Span	Standard Deviation (2)	Coefficient of variation(%)
51	0.0 - 1.8	0.0860	8.4
41	1.9 - 4.5	0.1318	4.2
21	4.6 - 9.0	0.1982	3.5
113	Overall	0.1302	

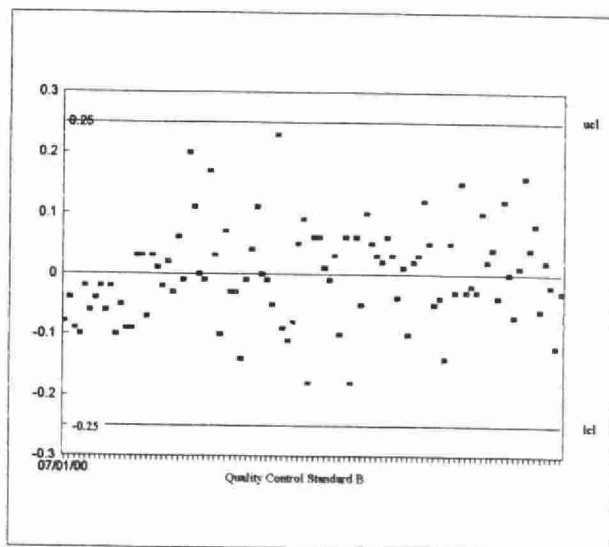
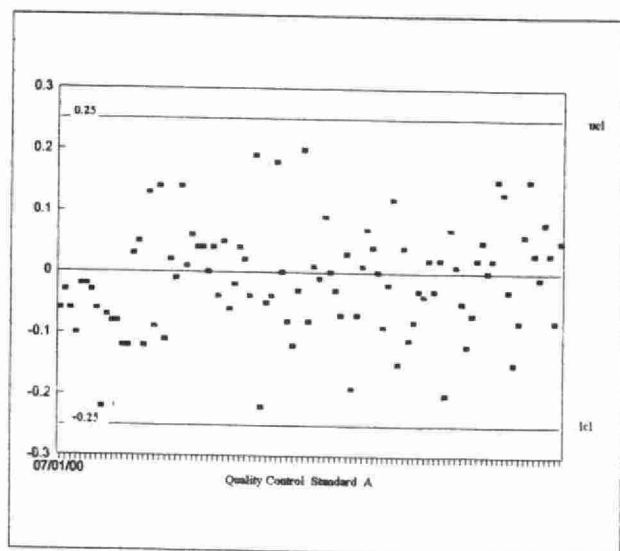
OTHER CHECKS:

	n	Mean	Standard Deviation (1)
5 Day Pure-DW Blank	49	0.192	0.1982
5 Day BOD Blank	49	0.166	0.1965

NOTES:

The final concentration of BOD in mg/L as O is determined by the oxygen depletion after 5 days at 20°C multiplied by a dilution and seed correction factor.

Oxygen Demand, Biochemical (mg/L as O)
Quality Control Data From 07/01/00 To 27/12/00
E3182



OXYGEN DEMAND, CHEMICAL

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/07/82
Method Reference No.	E3170	Reporting Unit	mg/L as O
LIMS Product Code	COD3170	Supervisor	P. Wilson
Sample Type/Matrix	Drinking Water, Ground Water, Surface Water		

SAMPLING:

Quantity Required	25 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Samples (10.0 mL) are mixed with an acidified potassium dichromate solution which contains mercuric sulphate to suppress chloride interference. After adding concentrated sulphuric acid containing silver sulphate as a catalyst, the mixture is digested in a mechanical-convection oven for 3 hours at 149°C. Analysis is completed by automated colourimetric measurement of trivalent chromium.

Approximate absorbance: 0.05 at the full scale level.

INSTRUMENTATION:

- Culture tubes with Teflon closures; mechanical-convection oven
- Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 600 nm.

REPORTING:

Maximum Significant Figures: 3	Current W value: 1	Current T value: 5
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CALIBRATION:

3 digested BL plus 3 digested standards

CONTROLS:

Calibration	2 digested standards, e.g. QCA
Drift	Undigested BL every 10 samples; standard plus BL at end of run
Recovery	2 digested standards, e.g. R1
Interference	Digested standard (40 mg/L as O) spiked with 50 mg/L Cl confirms suppression of chloride interference.

NOTES:

In order to retard sample decomposition the first reagent (acidified dichromate) is added as soon as possible at the laboratory. Analysis is scheduled for completion within the week. The recovery standard is a material known to be very difficult to digest. The expected recovery is approximately 85%, based on long term experience. We continue to use this material in spite of the poor recovery, because if the slightest problem exists with the digestion step, the recovery falls off sharply to approximately 10%.

OXYGEN DEMAND, CHEMICAL (E3170)

QUALITY CONTROL DATA FROM 11/01/00 TO 20/12/00

Full Scale: to 40.0 mg/L as O

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	35	40.0	40.29	0.29	0.7583
B:	35	10.0	9.43	-0.57	0.9837
A+B:	35	50.0	49.72	-0.28	1.3514
A-B:	35	30.0	30.85	0.85	1.1220

s.d.(AB)

S(between runs): 0.88

Sw(within run): 0.87

S/Sw: 1.11

On any given day the calibration is accepted if the calibration control values obtained lie within the ranges:

46.3 - 53.7 for A+B
27.2 - 32.8 for A-B

RECOVERIES:

Number of Data	Expected Concentration	Mean Concentration	Standard Deviation (1)
35	40	37.43	1.9447
35	10	8.24	0.6987

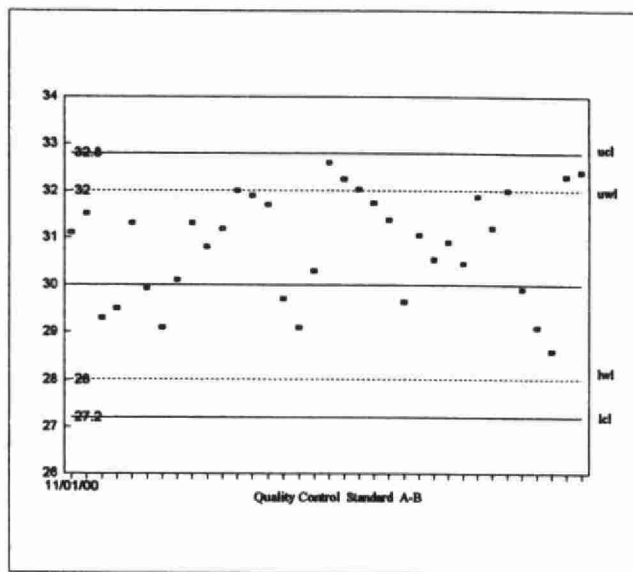
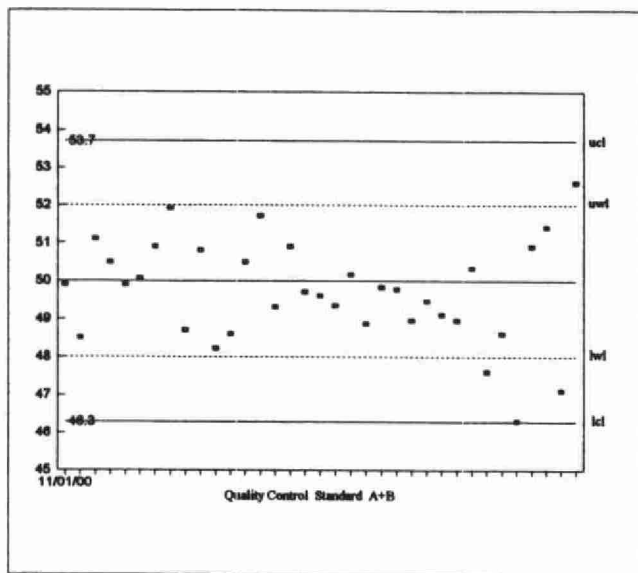
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
19	0 - 8	1.2698	29.9
37	9 - 20	1.3457	9.2
21	21 - 40	1.8161	7.0
77	Overall	1.4722	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Chloride Check	35	38.87	1.3374
Digested Blank	35	18.63	3.9563

OXYGEN DEMAND, CHEMICAL (mg/L as O)
QUALITY CONTROL DATA FROM 11/01/00 TO 20/12/00
E3170



OXYGEN DEMAND, CHEMICAL

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/07/82
Method Reference No.	E3246	Reporting Unit	mg/L as O
LIMS Product Code	COD3246	Supervisor	P. Wilson
Sample Type/Matrix	Raw Sewage, Industrial Waste, Ground Water, Leachate, Effluent, Sludge, Surface Water, Process Water		

SAMPLING:

Quantity Required	25 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Samples (10.0 mL) are mixed with an acidified potassium dichromate solution which contains mercuric sulphate to suppress chloride interference. After adding concentrated sulphuric acid containing silver sulphate as a catalyst, the mixture is digested in a mechanical-convection oven for 3 hours at 149°C. Analysis is completed by automated colourimetric measurement of trivalent chromium. Approximate absorbance: 0.6 at the full scale level.

INSTRUMENTATION:

-Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 600 nm.

REPORTING:

Maximum Significant Figures: 3	Current W value: 2	Current T value: 10
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CALIBRATION:

2 digested BL plus 4 digested standards

CONTROLS:

Calibration	2 digested standards, e.g. QCA
Drift	Undigested BL every 10 samples; standard plus BL at end of run
Recovery	2 digested standards, e.g. R1
Interference	Digested standard (50 mg/L as O) spiked with 900 mg/L Cl confirms suppression of chloride interference.

NOTES:

In order to retard sample decomposition the first reagent (acidified dichromate) is added as soon as possible at the laboratory. Analysis is scheduled for completion within the week. The recovery standard is a material known to be very difficult to digest. The expected recovery is approximately 85%, based on long term experience. We continue to use this material in spite of the poor recovery, because if the slightest

OXYGEN DEMAND, CHEMICAL (E3246)

QUALITY CONTROL DATA FROM 11/01/00 TO 18/12/00

Full Scale: to 500.0 mg/L as O

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	32	400	401.6	1.6	3.2882
B:	32	100	100.4	0.4	3.2181
A+B:	32	500	502.0	2.0	4.5786
A-B:	32	300	301.2	1.2	4.6230

s.d.(AB) S(between runs): 3.3

Sw(within run): 3.3 S/Sw:1.0

On any given day the calibration is accepted if the calibration control values obtained lie within the ranges:

477.5 - 522.5 for A+B
285.0 - 315.0 for A-B

RECOVERIES:

Number of Data	Expected Concentration	Mean Concentration	Standard Deviation (1)
32	390	385.1	8.7147
32	98	91.4	5.7197

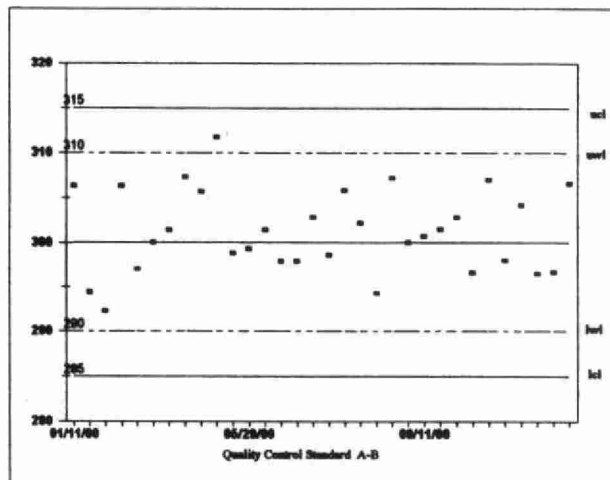
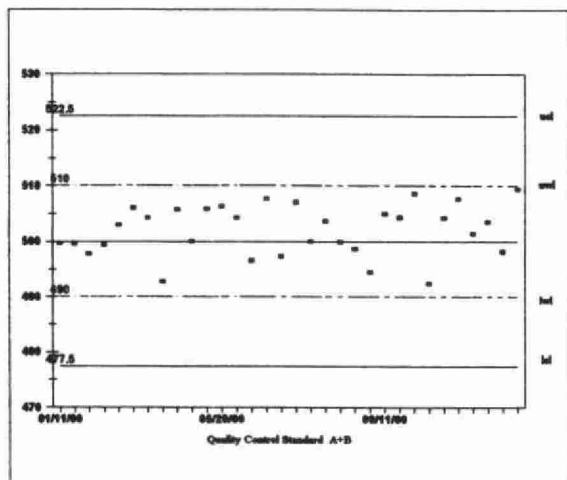
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
30	0 - 80	3.3480	8.3
17	81 - 200	5.2403	3.2
14	201 - 400	3.7030	1.4
61	Overall	4.0389	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Chloride Check	32	52.9	8.9949
Digested Blank	32	24.4	4.2498

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pH

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	09/07/80
Method Reference No	E3218	Reporting Units	Dimensionless
LIMS Product Code	PHALCO3218, CONDPH3218	Supervisor	P. Wilson
Sample Type/Matrix	Sludge, Effluent, Industrial Waste, Raw Sewage, Drinking Water, Ground Water, Leachate, Precipitation, Surface Water		

SAMPLING:

Quantity Required	50 mL
Container	Glass or Plastic

ANALYTICAL PROCEDURE:

pH is directly measured on a stirred sample (20.0 mL) at room temperature. Stirring rate, tube size, degree of electrode immersion, and room temperature range are uniform for all samples and standards. Total fixed endpoint alkalinity, and conductivity are determined simultaneously.

INSTRUMENTATION:

Automated modular titration system with microcomputer control and data processing software.

REPORTING:

Maximum Significant Figures: 3

CALIBRATION:

2 standard buffers covering the pH range of 4 to 9

CONTROLS:

Calibration	2 QC standards e.g. QCA
Drift	In run standards throughout the run (diluted tap water 50% V/V)

pH (E3218)

QUALITY CONTROL DATA FROM 05/01/00 TO 19/12/00

Analytical Range: to 14.00 Dimensionless

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	110	7.413	7.4134	0.0004	0.0111
B:	110	4.45	4.5348	0.0848	0.0365
A+B:		11.86	11.9480	0.0882	0.0361
A-B:		2.96	2.8786	-0.0814	0.0401

s.d.(AB) S(between runs): 0.027 Sw(within run): 0.028 S/Sw: 0.95

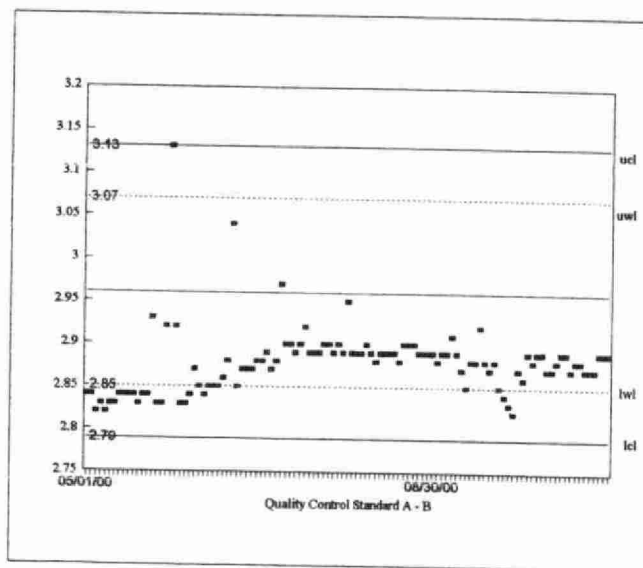
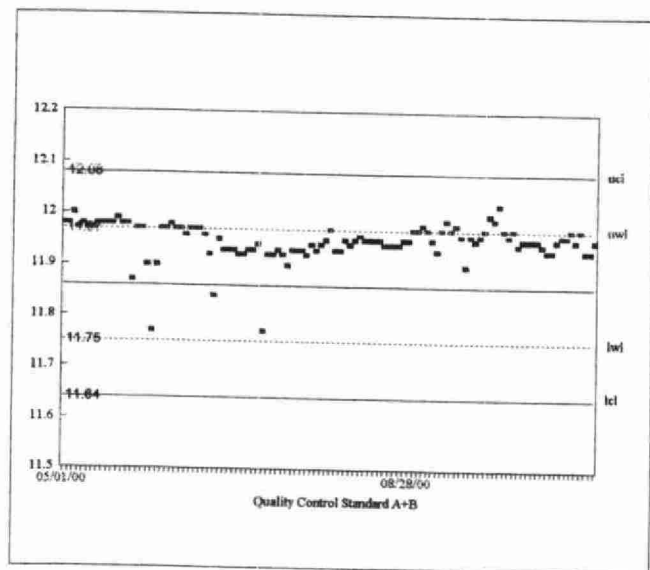
On any given day the calibration is accepted if the values obtained lie within the ranges:

11.64 - 12.08 for A+B
2.79 - 3.13 for A-B

DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
32	1.00 - 7.00	0.0422	0.7
141	7.01 - 8.00	0.0622	0.8
140	8.01 - 12.00	0.0452	0.5
313	Overall	0.0533	

pH
Quality Control Data from 05/01/00 to 19/12/00
E3218



pH

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/05/79
Method Reference No	E3248	Reporting Units	Dimensionless
LIMS Product Code	PHACD3248, PH3248	Supervisor	P. Wilson
Sample Type/Matrix	Effluent, Industrial Waste, Raw Sewage, Drinking Water, Leachate, Precipitation, Surface Water		

SAMPLING:

Quantity Required	15 mL
Container	Glass or Plastic

ANALYTICAL PROCEDURE:

pH is directly measured on a stirred sample (15.0 mL) at room temperature. Stirring rate, tube size, degree of electrode immersion, and room temperature range are uniform for all samples and standards. Total fixed endpoint acidity and Gran acidity can be determined simultaneously if the sample volume exceeds 50 mL.

INSTRUMENTATION:

Automated modular titration system with microcomputer control and data processing software.

REPORTING:

Maximum Significant Figures: 3

CALIBRATION:

2 standard buffers covering the pH range of 4 to 9

CONTROLS:

Calibration	LTBL plus 2 standards, e.g. QCA
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NOTES:

A new automated system was introduced in Sept' 96.

pH (E3248)

QUALITY CONTROL DATA FROM 11/01/00 TO 25/10/00

Analytical Range: to 14.00 Dimensionless

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	15	4.45	4.420	-0.030	0.0501
B:	15	3.75	3.706	-0.044	0.0290
A+B:		8.20	8.143	-0.057	0.0752
A-B:		0.70	0.721	0.021	0.0389

s.d.(AB) S(between runs): 0.03

S/Sw:(within run): 0.02

S/Sw: 1.4

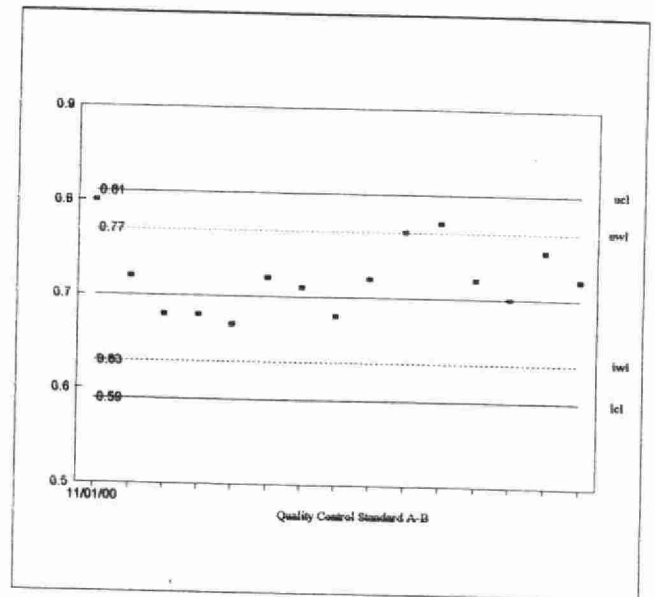
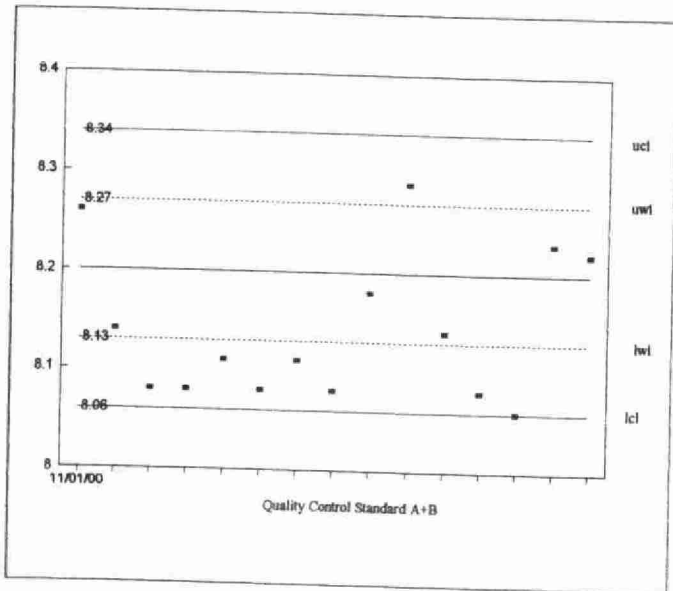
On any given day the calibration is accepted if the values obtained lie within the ranges:

8.06 - 8.34 for A+B
0.59 - 0.81 for A-B

DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
3	2.50 - 4.00	0.0082	0.2
15	4.01 - 5.00	0.0171	0.4
14	5.01 - 8.00	0.0943	1.4
32	Overall	0.0635	

pH
 QUALITY CONTROL DATA FROM 11/01/00 TO 25/10/00
 E3248



PHENOLICS, REACTIVE

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/04/74
Method Reference No.	E3179	Reporting Unit	µg/L as Phenol
LIMS Product Code	PHEN3179	Supervisor	P. Wilson
Sample Type/Matrix	Ground Water, Surface Water, Effluent, Drinking Water, Leachate, Raw Sewage, Industrial Waste, Process Water, Precipitation		

SAMPLING:

Quantity Required	250 mL
Container	Glass, (Phenol bottle with white cap containing preservative is available)
Preservative	Sulfuric acid to pH 1.5 - 2

ANALYTICAL PROCEDURE:

Samples are automatically distilled from an acid media, and reactive phenolics in the distillate are determined colourimetrically by formation of an antipyrene dye through reactions with 4-aminoantipyrene and potassium ferricyanide.

Approximate absorbance: 0.03 at the full scale level.

INSTRUMENTATION:

Basic automated modular continuous flow system plus a distillation module. Colourimetric measurement is through a 5.0 cm. light path at 505 nm.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.2	Current T value: 1
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CALIBRATION:

BL plus 2 standards

CONTROLS:

Calibration	LTBL plus 2 standards, e.g. QCA (see note)
Drift	BL ,standard ,BL every 10 samples

NOTES:

An additional Quality Control Standard (QCC) was added to the method in March 1997.

PHENOLICS, REACTIVE

QUALITY CONTROL DATA FROM 07/01/00 TO 29/12/00

Laboratory Unit: Colourimetry

Full Scale: to 50.0 µg/L as Phenol

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	46	40	40.0361	0.0361	0.3672
B:	46	10	10.1261	0.1261	0.1632
C:	46	5	5.0793	0.0793	0.1253
A+B:		50	50.1622	0.1622	0.4041
A-B:		30	29.9100	-0.0900	0.3907
B+C:		15	15.2054	0.2054	0.2471
B-C:		5	5.0467	0.0467	0.1475

s.d.(AB) S(between runs):0.28

s.d.(BC) S(between runs):0.15

Sw(within run): 0.28 S/Sw:1.0

Sw(within run): 0.11 S/Sw:1.4

On any given day the calibration is accepted if the calibration control values obtained lie within the ranges:

47.8	-	52.2	for	A+B
28.3	-	31.7	for	A-B
14.0	-	16.0	for	B+C
4.25	-	5.75	for	B-C

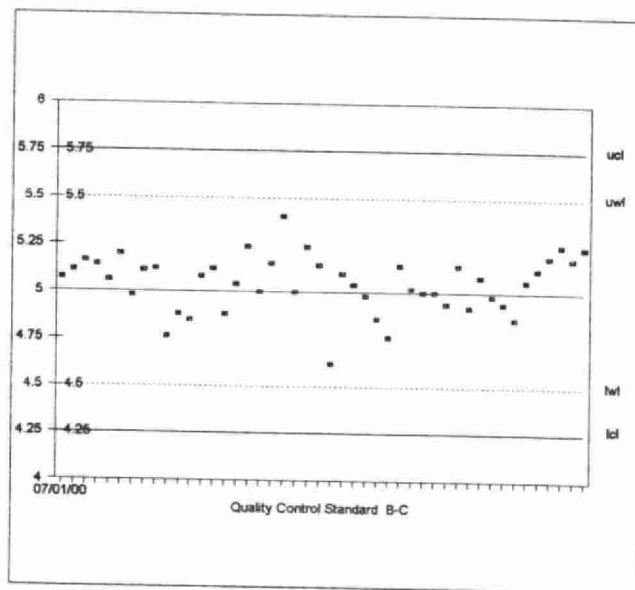
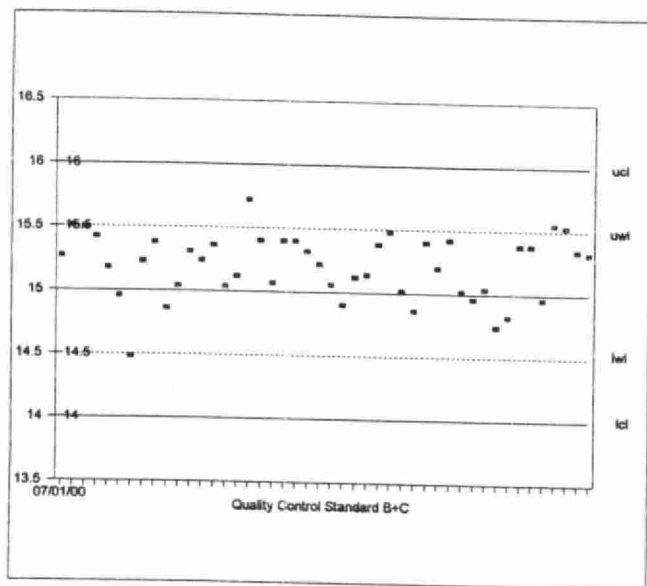
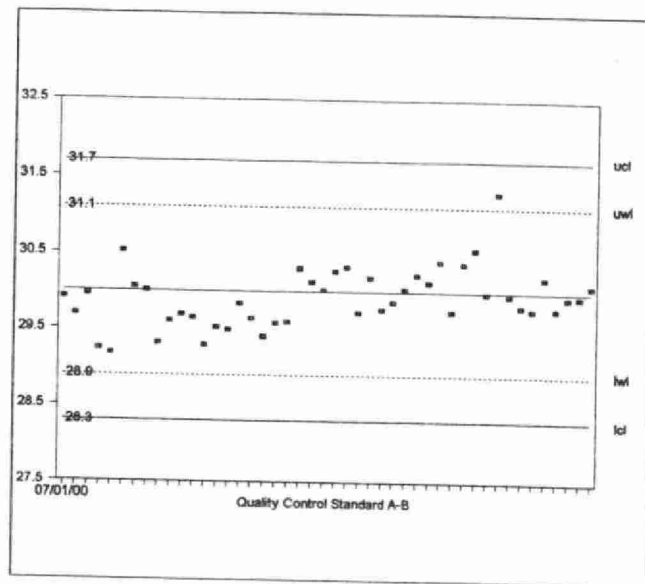
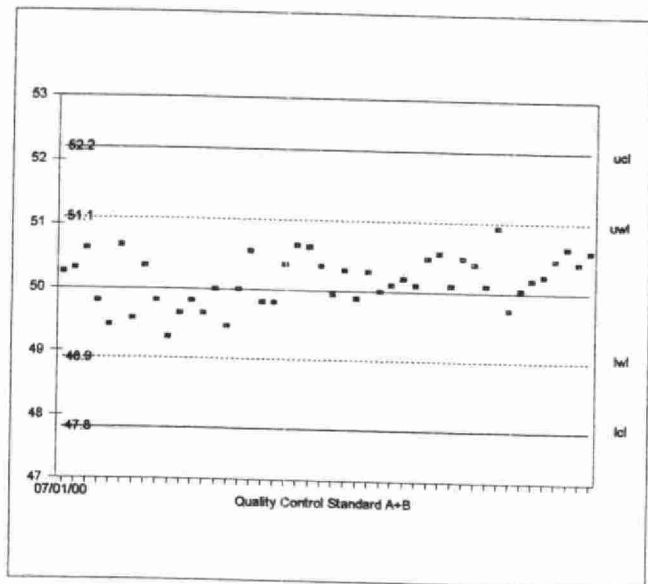
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
108	0.00 - 5.0	0.1386	23.3
6	5.1 - 10.0	0.2434	3.2
9	10.1 - 25.0	0.3038	2.0
1	25.1 - 50.0	N.A.	N.A.
124	Overall	0.5049	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	46	0.0196	0.1116

Phenolics, Reactive ($\mu\text{g/L}$ as Phenol)
Quality Control Data From 07/01/00 To 29/12/00
E3179



PHOSPHOROUS, REACTIVE ortho-PHOSPHATE

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/04/79
Method Reference No.	E3364	Reporting Unit	mg/L as P
LIMS Product Code	DISNUT3364	Supervisor	P. Wilson
Sample Type/Matrix	Dried Sludge, Sediment, Soil, Vegetation, Drinking Water, Ground Water, Surface Water		

SAMPLING:

Quantity Required	10 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Ortho-phosphate is determined on the supernatant of a settled sample by formation of the reduced phospho-antimonyl-molybdate complex using ascorbic acid as the reducing agent.

Approximate absorbance: 0.2 at the full scale level.

Ammonia plus ammonium, nitrite, and nitrate plus nitrite are determined simultaneously.

INSTRUMENTATION:

Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 880 nm using IR sensitive phototube.

Data capture, reduction, and processing via a multi-stage microcomputer system.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.0005	Current T value: 0.0025
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CALIBRATION:

BL plus 7 standards

CONTROLS:

Calibration	LTBL plus 3 standards, e.g. QCA
Drift	BL ,standard ,and BL after every 10 samples

NOTES:

The HP data capture / processing system was replaced by Labtronics in August 1999.

PHOSPHORUS, REACTIVE ortho-PHOSPHATE (E3364)

QUALITY CONTROL DATA FROM 05/01/00 TO 05/12/00

Laboratory Unit: Colourimetry

Full Scale: to 0.100 mg/L as P

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	104	0.080	0.0796	-0.0004	0.0012
B:	104	0.040	0.0399	-0.0001	0.0010
C:	104	0.008	0.0079	-0.0001	0.0007
A+B:	104	0.120	0.1196	-0.0004	0.0017
A-B:	104	0.040	0.0396	-0.0004	0.0014
B+C:	104	0.048	0.0479	-0.0001	0.0013
B-C:	104	0.032	0.0321	0.0001	0.0012

s.d.(AB)

S(between runs):0.0011

Sw(within run):

0.0011

S/Sw: 1.1

s.d.(BC)

S(between runs):0.0009

Sw(within run):

0.0008

S/Sw: 1.1

The calibration is accepted if the calibration control values obtained lie within the ranges:

0.1152 - 0.1248 for A+B
0.0364 - 0.0436 for A-B
0.0448 - 0.0512 for B+C
0.0296 - 0.0344 for B-C

DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
213	0.000 - 0.010	0.0009	33.9
25	0.011 - 0.020	0.0016	11.2
28	0.021 - 0.050	0.0022	6.9
16	0.051 - 0.100	0.0023	3.2
282	Overall	0.0013	

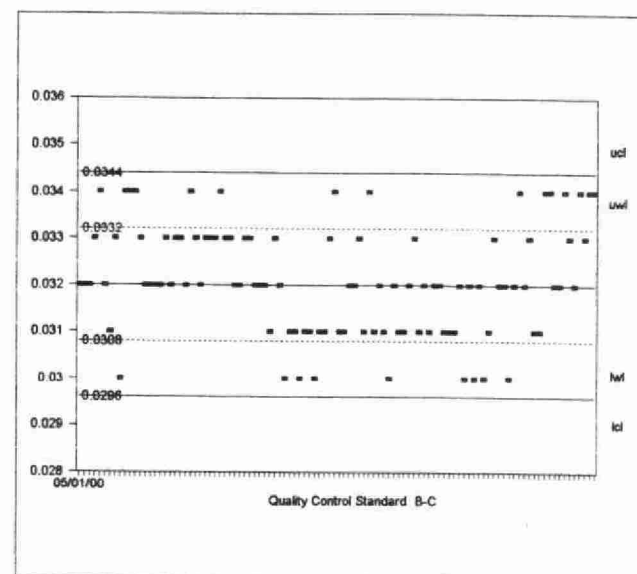
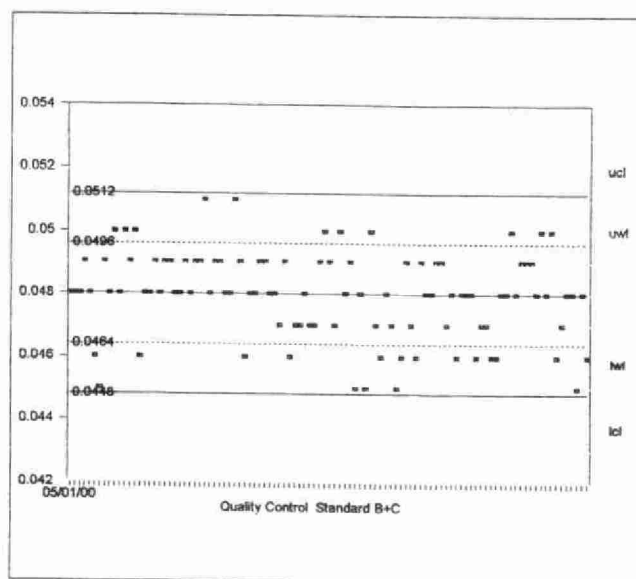
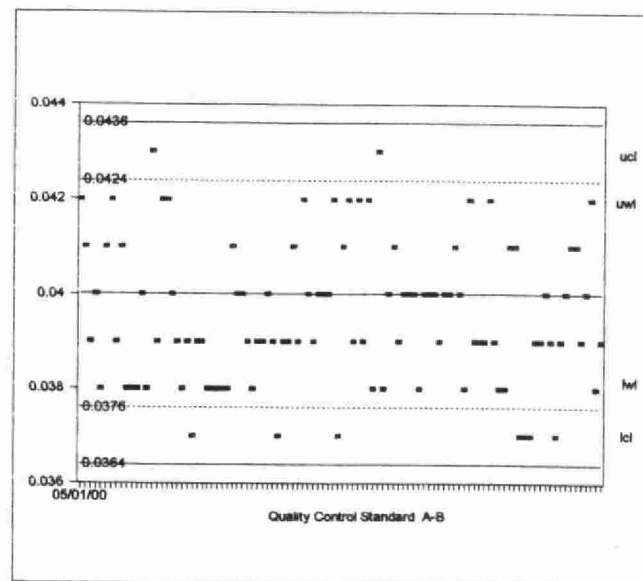
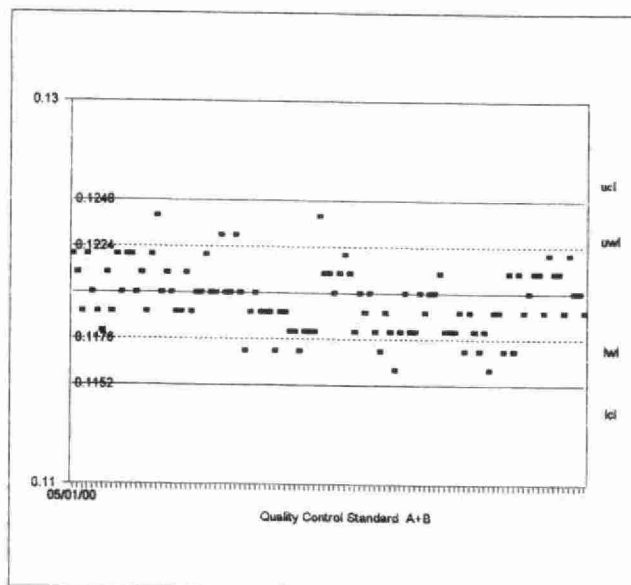
OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	104	0.00056	0.0011

PHOSPHOROUS, REACTIVE ortho-PHOSPHATE (mg/L as P)

QUALITY CONTROL DATA FROM 05/01/00 TO 05/12/00

E3364



PHOSPHORUS, REACTIVE ortho-PHOSPHATE

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/04/79
Method Reference No	E3366	Reporting Unit	mg/L as P
LIMS Product Code	DISNUT3366	Supervisor	P.Wilson
Sample Type/Matrix	Sludge, Effluent, Raw Sewage, Industrial Waste, Process Water, Leachate, Drinking Water, Ground Water, Surface Water.		

SAMPLING:

Quantity Required	10 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Ortho-phosphate is determined on the supernatant of a settled sample by formation of the reduced phospho-antimonyl-molybdate complex using ascorbic acid as the reducing agent.

Approximate absorbance: 0.5 at the full scale level.

Ammonia plus ammonium, nitrite, and nitrate plus nitrite are determined simultaneously.

INSTRUMENTATION:

Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 880 nm using IR sensitive phototube.

Data capture, reduction, and processing via a multi-stage microcomputer system.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.02	Current T value: 0.1
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CALIBRATION:

BL plus 7 standards

CONTROLS:

Calibration	LTBL plus 3 standards, e.g. QCA
Drift	BL, standard and BL every 10 samples

NOTES:

The HP capture / processing system was replaced by Labtronics in October 1999.

PHOSPHORUS, REACTIVE ortho-PHOSPHATE (E3366)

QUALITY CONTROL DATA FROM 06/01/00 TO 21/12/00

Laboratory Unit: Colourimetry

Full Scale: to 10.0 mg/L as P

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	78	8.00	8.002	0.002	0.0461
B:	78	4.00	4.008	0.008	0.0368
C:	78	0.800	0.802	0.002	0.0205
A+B:		12.0	12.010	0.001	0.0582
A-B:		4.00	3.994	-0.006	0.0597
B+C:		4.80	4.810	0.001	0.0445
B-C:		3.20	3.206	0.006	0.0396

s.d.(AB) S(between runs):0.0417

s.d.(BC) S(between runs):0.0298

Sw(within run): 0.0422

Sw(within run): 0.0280

S/Sw: 0.99

S/Sw: 1.06

The calibration is accepted if the calibration control values obtained lie within the ranges:

11.71	-	12.29	for	A+B
3.78	-	4.22	for	A-B
4.66	-	4.94	for	B+C
3.09	-	3.31	for	B-C

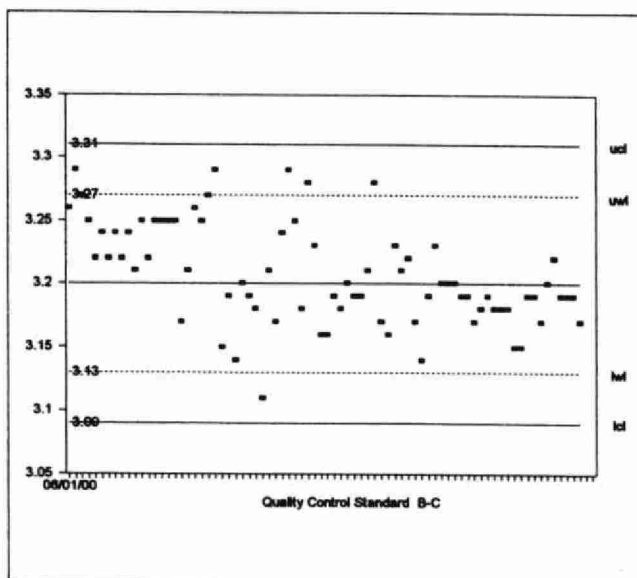
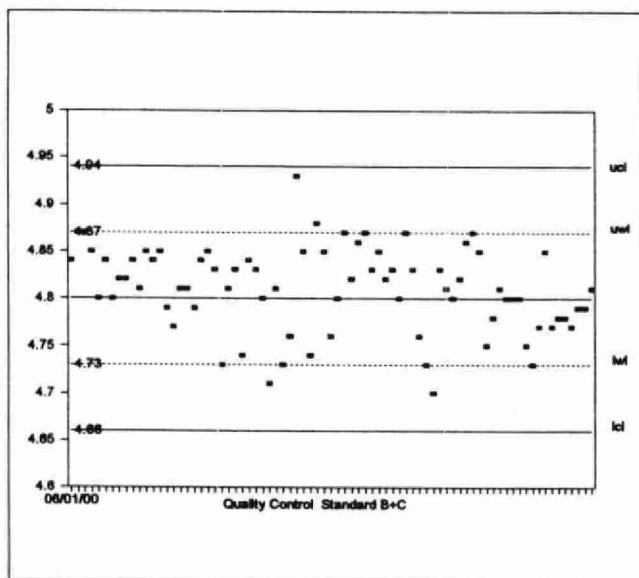
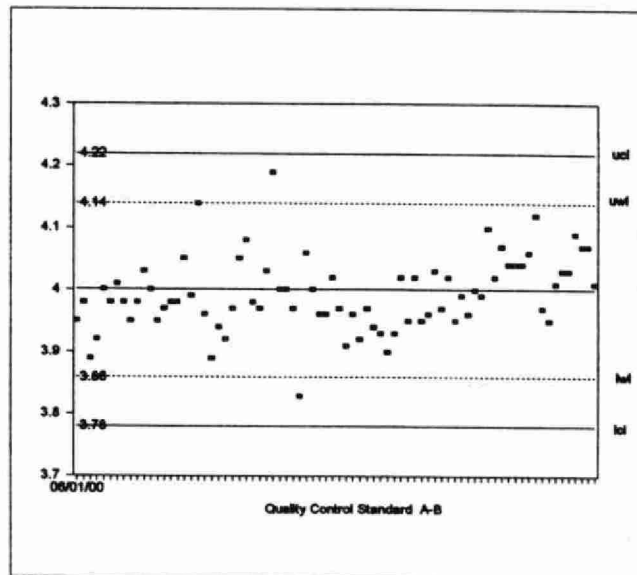
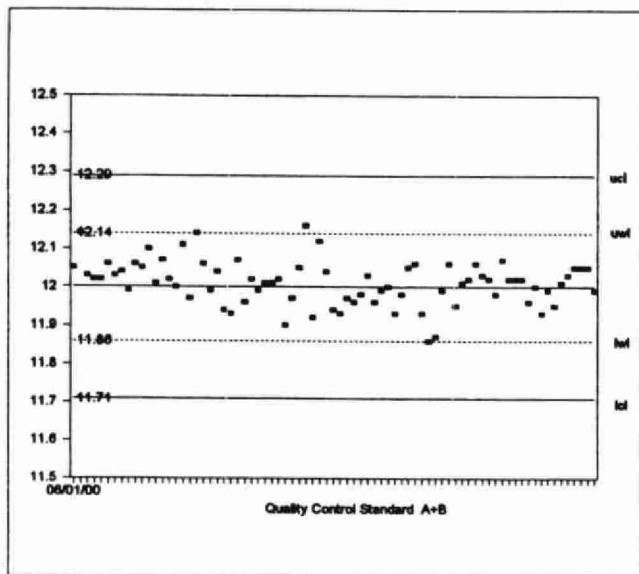
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
194	0.000 - 1.00	0.0215	14.4
10	1.01 - 2.00	0.0356	2.3
14	2.01 - 5.00	0.0494	1.7
1	5.01 - 10.0	N.A.	N.A.
219	Overall	0.0250	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	78	0.0429	0.0285

PHOSPHORUS, REACTIVE ortho-PHOSPHATE (mg/L as P)
QUALITY CONTROL DATA FROM 06/01/00 TO 21/12/00
E3366



PHOSPHORUS, TOTAL

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	Mar '89
Method Reference No.	E3116	Reporting Unit	mg/g as P
LIMS Product Code	TNP3116	Supervisor	P. Wilson
Sample Type/Matrix	Soil, Sediment, Dried Sludge		

SAMPLING:

Quantity Required	0.08 to 0.4 g
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Phosphorus compounds are converted to simple inorganic forms by dissolution of the samples in hot sulphuric acid and potassium persulphate. Potassium persulphate is added later in the digestion to raise the boiling point and to provide a highly oxidizing environment to decompose the more resistant organic matter. The digestate is filtered and the filtrate is analyzed using an automated colourimetric system.

INSTRUMENTATION:

Hot plate

Basic automated modular continuous flow system : Colourimetric measurement is through a 5 cm. light path at 660 nm.

Data capture, and processing via a microcomputer system

REPORTING:

Maximum Significant Figures: 2 decimal places	Current W value: 0.02	Current T value: 0.10
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CALIBRATION:

3 High and 2 Low Calibration Standards

CONTROLS:

Calibration	In house composite B-Soil/sediment, plus QC Soils/Sediment (RS92)
Drift	4 BL's per run; high and low calibration standard at the end of the run
Recovery	1 digested BL plus 4 digested standards

NOTES:

System is calibrated with undigested standards.

PHOSPHORUS, TOTAL (E3116)

QUALITY CONTROL DATA FROM 12/01/00 TO 04/12/00

Full Scale: 2 mg/g as P

QUALITY CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
RS92 In House Soil Composite	36	0.47	0.442	-0.028	0.0464
RSM-2781 -Reference Standard Material	38	25.5	23.432	-2.068	1.2086

The run is accepted if the control values obtained lie within the ranges:

0.40	-	0.54	for	RS92
23.3	-	25.1	for	RSM-2781

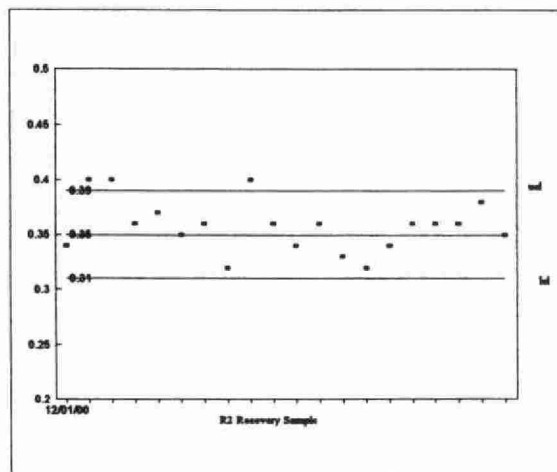
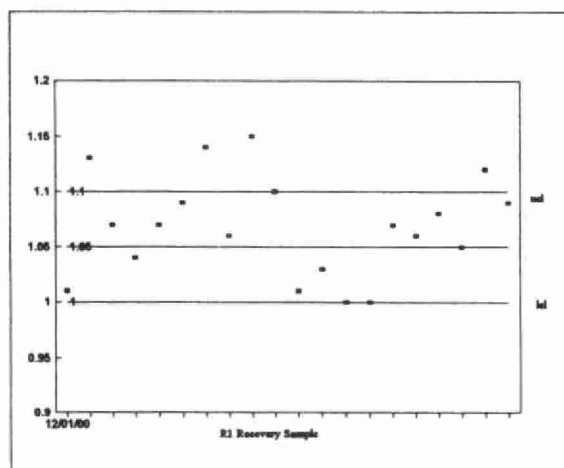
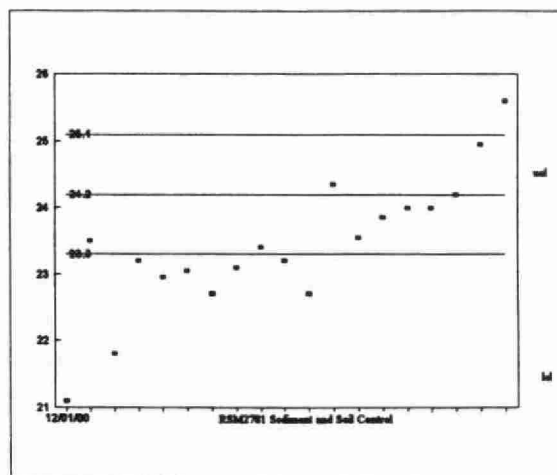
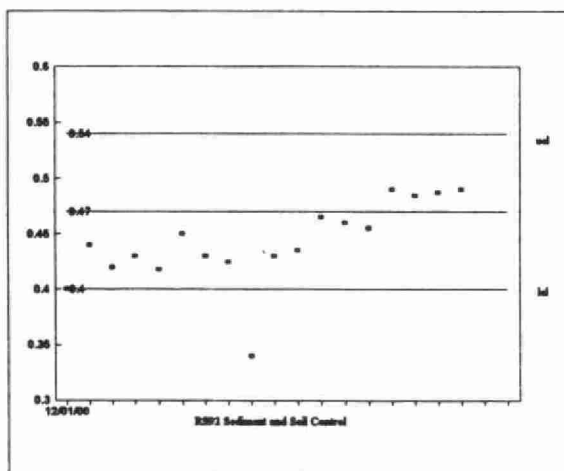
Recovery Standards

	n	Expected Concentration	Mean Concentration	Standard Deviation (1)
R1	20	1.05	1.069	0.0455
R2	20	0.35	0.358	0.0238

DUPLICATES: (Sediment/Soils)

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
5	0.00 - 0.50	0.0339	8.7
17	0.6 - 1.0	0.0411	5.2
16	1.1 - 2.5	0.1510	9.1
7	2.6 - 5.0	0.2300	6.3
45	Overall	0.1308	

PHOSPHORUS , TOTAL (mg/L as P)
QUALITY CONTROL DATA FROM 12/01/00 TO 04/12/00
E3116



PHOSPHORUS, TOTAL

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	Mar '89
Method Reference No.	E3118	Reporting Unit	mg/g as P
LIMS Product Code	TNP3118	Supervisor	P. WILSON
Sample Type/Matrix	Vegetation, Moss Bag		

SAMPLING:

Quantity Required	0.02 to 0.04 g
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Phosphorus compounds are converted to simple inorganic forms by dissolution of the samples in hot sulphuric acid and potassium persulphate. Potassium persulphate is added later in the digestion to raise the boiling point and to provide a highly oxidizing environment to decompose the more resistant organic matter. The digestate is analyzed using an automated colourimetric system.

INSTRUMENTATION:

Hot plate.

Basic automated modular continuous flow system : Colourimetric measurement is through a 5 cm. light path at 660 nm.

Data capture, and processing via a microcomputer system.

REPORTING:

Maximum Significant Figures: 2 decimal places	Current W value: 0.02	Current T value: 0.10
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CALIBRATION:

3 High and 2 Low Calibration Standards

CONTROLS:

Calibration	In house composite A-VEG, plus QC VEG (Pine Needles)
Drift	4 BL's per run; high and low calibration standard at the end of the run
Recovery	1 digested BL plus 4 digested standards

NOTES:

System is calibrated with undigested standards.

There is not enough data to report QC for 2000.

PHOSPHORUS, TOTAL

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/04/79
Method Reference No	E3367	Reporting Unit	mg/L as P
LIMS Product Code	TOTNUT3367	Supervisor	P. Wilson
Sample Type/Matrix	Precipitation, Drinking Water, Ground Water , Surface Water		

SAMPLING:

Quantity Required	50 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Samples are digested in a sulphuric acid-mercuric oxide-potassium sulphate media using three block digesters kept at 180°C, 210°C and 360°C. The pH of the digestate is adjusted in-line and then orthophosphate is determined by formation of the reduced phospho-antimonyl-molybdate complex using ascorbic acid as the reducing agent.

Approximate absorbance: 0.4 at the full scale level.

Total Kjeldahl nitrogen is determined simultaneously.

INSTRUMENTATION:

Three Block digesters

Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 880 nm using appropriate phototube.

Data capture, reduction, and processing via a multi-stage microcomputer system

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.002	Current T value: 0.01
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CALIBRATION:

BL plus 7 undigested standards

CONTROLS:

Calibration	LTBL plus 3 undigested standards, e.g. QCA
Drift	BL , undigested standard , BL every 10 samples
Recovery	3 digested BL plus 3 digested standards in duplicate, e.g. R1

NOTE:

The HP capture / processing system was replaced by Labtronics in May 1999.

PHOSPHORUS, TOTAL (E3367)

QUALITY CONTROL DATA FROM 06/01/00 TO 29/12/00

Full Scale: to 0.200 mg/L as P

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	103	0.160	0.1613	0.0013	0.0011
B:	103	0.080	0.0804	0.0004	0.0010
C:	103	0.016	0.0160	0.0000	0.0008
A+B:	103	0.240	0.2417	0.0017	0.0017
A-B:	103	0.080	0.0809	0.0009	0.0012
B+C:	103	0.096	0.0965	0.0005	0.0016
B-C:	103	0.064	0.0644	0.0004	0.0008

s.d.(AB) S(between runs): 0.00104 Sw(within run): 0.00087 S/Sw: 1.2
s.d.(BC) S(between runs): 0.00089 Sw(within run): 0.00053 S/Sw: 1.7

The calibration is accepted if the calibration control values obtained lie within the ranges:

0.2332 - 0.2468 for A+B
0.0749 - 0.0851 for A-B
0.092 - 0.100 for B+C
0.061 - 0.067 for B-C

RECOVERIES:

Number of Data	Expected Concentration	Mean Concentration	Standard Deviation (1)
103	0.140	0.1402	0.0044
103	0.084	0.0840	0.0031
103	0.029	0.0290	0.0021

DUPLICATES:

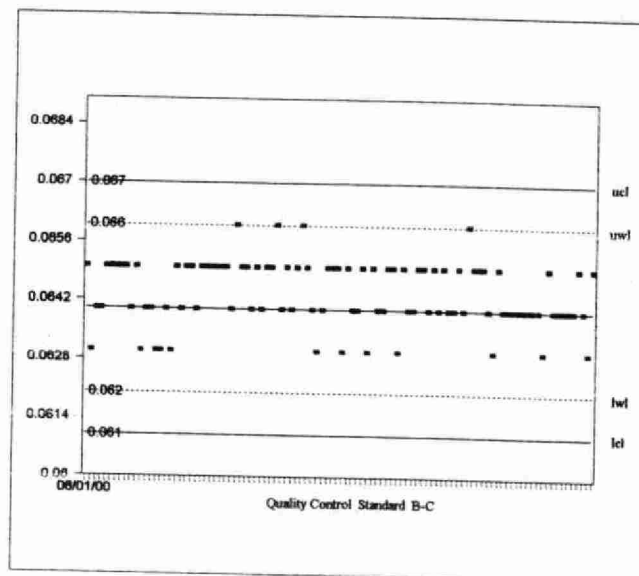
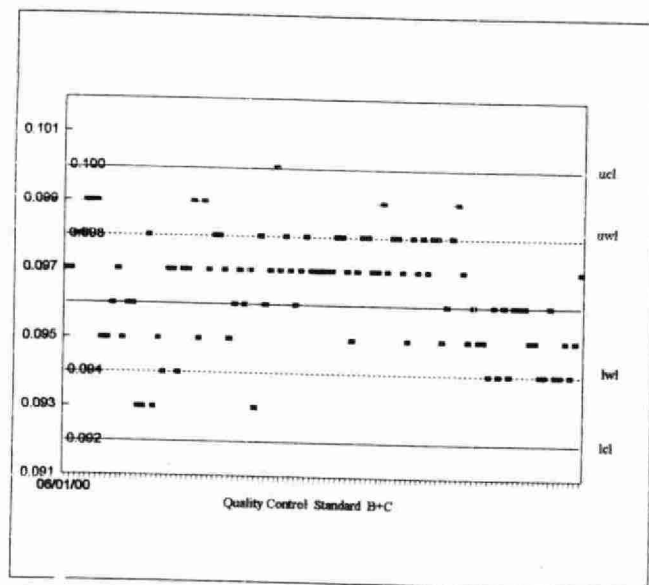
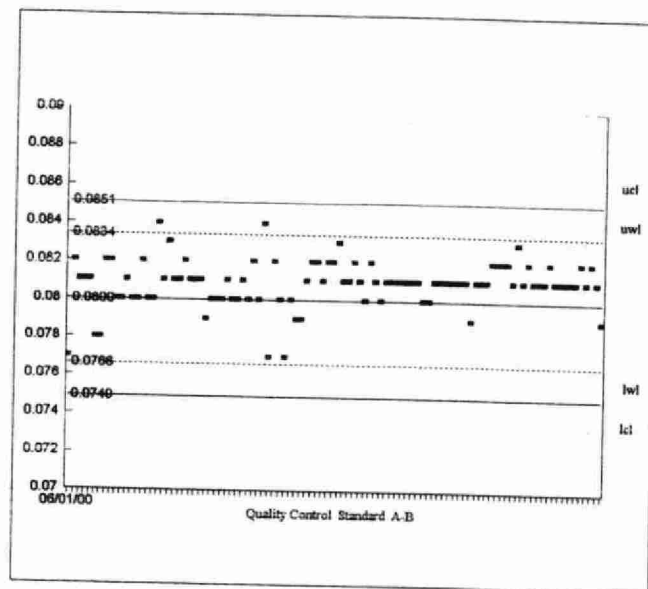
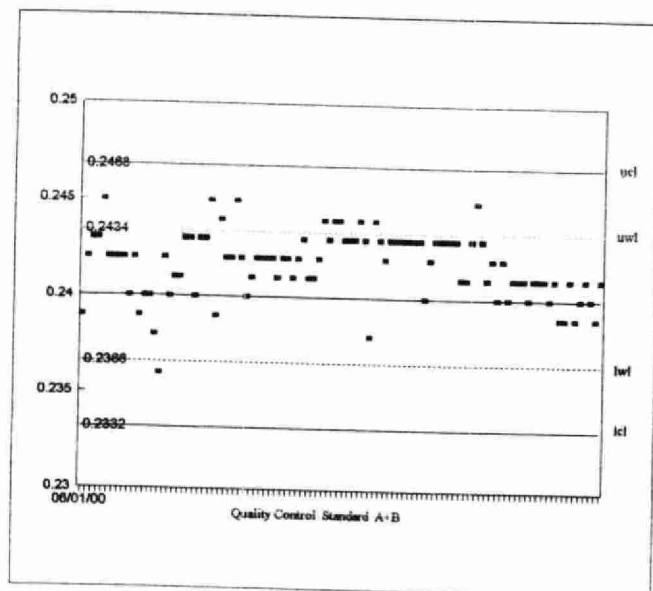
n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
191	0.000 - 0.020	0.0054	55.4
41	0.021 - 0.040	0.0102	35.5
32	0.041 - 0.100	0.0146	23.7
9	0.101 - 0.200	0.0068	5.2
273	Overall	0.0079	

PHOSPHORUS, TOTAL (E3367) cont'd

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	103	0.0004	0.0009
Digested Blank	103	0.0023	0.0016

PHOSPHOROUS, TOTAL (mg/L as N)
QUALITY CONTROL DATA FROM 06/01/00 TO 29/12/00
E3367



PHOSPHORUS, TOTAL

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/04/79
Method Reference No.	E3368	Reporting Unit	mg/L as P
LIMS Product Code	TOTNUT3368	Supervisor	J. McBride
Sample Type/Matrix	Sludge, Raw Sewage, Industrial Waste, Drinking Water, Effluent, Ground Water, Process Water, Leachate, Surface Water.		

SAMPLING:

Quantity Required	50 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Samples are digested in a sulphuric acid-mercuric oxide-potassium sulphate media using three block digestors kept at 180°C, 210°C and 360°C. The pH of the digestate is adjusted in-line and then orthophosphate is determined by formation of the reduced phospho-antimonyl-molybdate complex using ascorbic acid as the reducing agent.

Approximate absorbance: 0.8 at the full scale level.

Total Kjeldahl Nitrogen is determined simultaneously.

INSTRUMENTATION:

3-Block digesters

Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 880 nm using an IR sensitive phototube. Data capture, reduction, and processing via a multi-stage microcomputer system.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.02	Current T value: 0.10
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CALIBRATION:

BL plus 7 standards

CONTROLS:

Calibration	LTBL plus 3 standards, e.g. QCA
Drift	BL every 10 samples; undigested standard every 20 samples
Recovery	3 digested BL plus 3 digested standards in duplicate, e.g. R1

NOTES:

System is calibrated with undigested standards.

The HP capture / processing system was replaced by Labtronics in April 1999

PHOSPHORUS, TOTAL (E3368)

QUALITY CONTROL DATA FROM 18/01/00 TO 21/12/00

Full Scale: to 10.0 mg/L as P

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	49	8.0	8.009	0.009	0.0325
B:	49	4.0	4.008	0.008	0.0194
C:	49	0.8	0.801	0.001	0.0115
A+B:		12.0	12.017	0.017	0.0419
A-B:		4.0	4.001	0.001	0.0333
B+C:		4.8	4.809	0.009	0.0239
B-C:		3.2	3.206	0.006	0.0212

s.d.(AB) S(between runs): 0.0268 Sw(within run): 0.0235 S/Sw: 1.1
s.d.(BC) S(between runs): 0.0160 Sw(within run): 0.0150 S/Sw: 1.1

The calibration is accepted if the calibration control values obtained lie within the ranges:

11.87 - 12.13 for A+B
3.903 - 4.097 for A-B
4.732 - 4.868 for B+C
3.149 - 3.251 for B-C

RECOVERIES:

Number of Data	Expected Concentration	Mean Concentration	Standard Deviation (1)
49	7	6.85	0.1444
49	4.2	4.13	0.0835
49	1.4	1.38	0.0344

DUPLICATES:

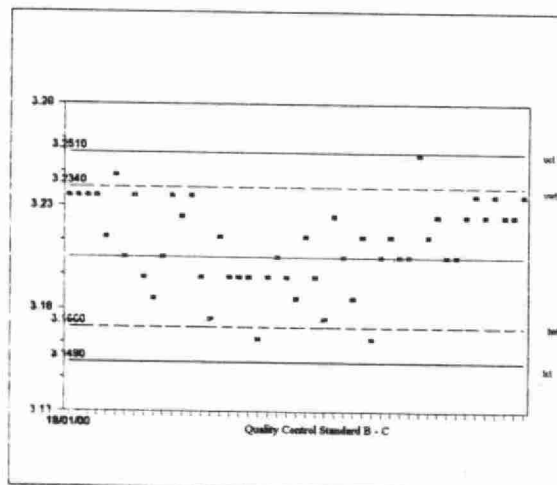
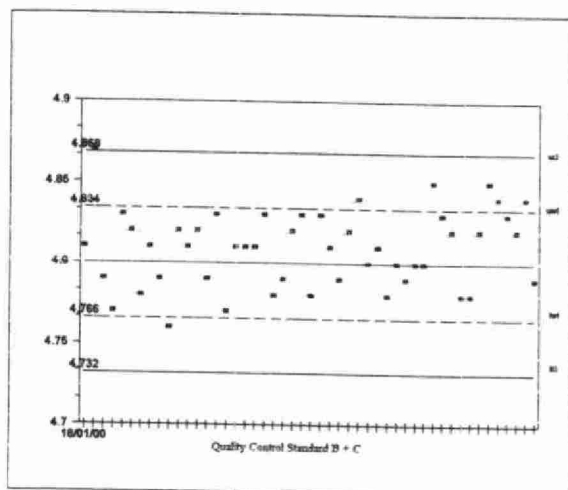
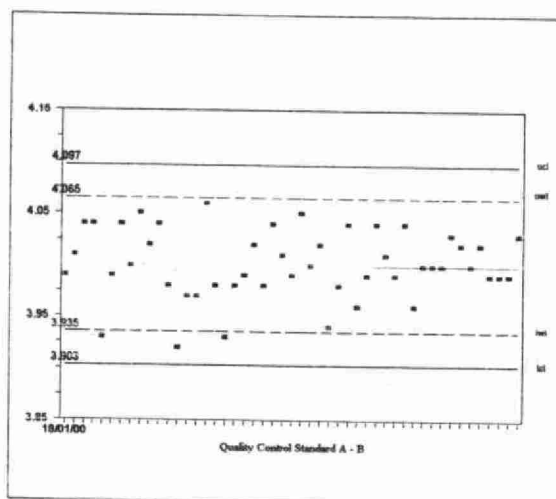
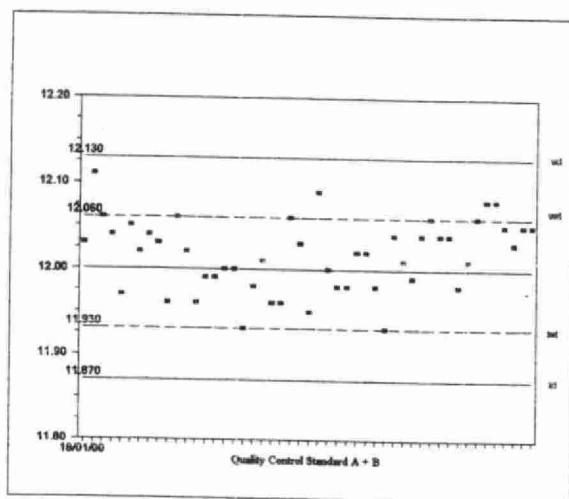
n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
113	0.0 - 1.0	0.0690	38.1
4	1.1 - 2.0	0.0579	3.4
18	2.1 - 5.0	0.1703	5.3
2	5.1 - 10.0	N.A.	N.A.
137	Overall	0.1097	

PHOSPHORUS, TOTAL cont:

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	49	0.006	0.0170
Digested Blank	49	0.010	0.0169

PHOSPHORUS, TOTAL (mg/L as P)
 QUALITY CONTROL DATA FROM 18/01/00 TO 21/12/00
 E3368



SILICON, REACTIVE SILICATES

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/02/75
Method Reference No.	E3370	Reporting Unit	mg/L as Si
LIMS Product Code	DCSI3370	Supervisor	P.Wilson
Sample Type/Matrix	Effluent, Industrial Waste, Process Water, Raw Sewage, Drinking Water Ground Water, Leachates, Precipitation, Surface Water		

SAMPLING:

Quantity Required	10 mL
Container	Plastic

ANALYTICAL PROCEDURE:

Reactive silicates are determined by formation of a reduced molybdo-silicate complex at pH 1.6, using ascorbic acid as the reducing agent, and oxalic acid to suppress phosphate interference.

Approximate absorbance: 0.7 at the full scale level.

Dissolved inorganic and dissolved organic carbon are determined simultaneously.

INSTRUMENTATION:

Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 660 nm. Data capture, reduction, and processing via a multi-stage microcomputer system.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.02	Current T value: 0.10
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CALIBRATION:

BL plus 7 standards

CONTROLS:

Calibration	LTBL plus 3 standards, e.g., QCA
Drift	BL, standard and BL every 10 samples.

NOTES:

December 1998: The HP data capture/processing system was replaced by Labtronics.

SILICON, REACTIVE SILICATES (E3370)

QUALITY CONTROL DATA FROM 07/01/00 TO 18/12/00

Full Scale: to 10.0 mg/L as Si

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	69	8.00	8.034	0.034	0.0623
B:	69	2.00	2.015	0.015	0.0160
C:	69	0.50	0.486	-0.014	0.0083
A+B:		10.00	10.050	0.050	0.0744
A-B:		6.00	6.019	0.019	0.0525
B+C:		2.50	2.501	0.001	0.0218
B-C:		1.50	1.529	0.029	0.0131

s.d.(AB) S(between runs): 0.044

Sw(within run): 0.036

S/Sw: 1.2

s.d.(BC) S(between runs): 0.013

Sw(within run): 0.009

S/Sw: 1.4

The calibration is accepted if the calibration control values obtained lie within the ranges:

9.66	-	10.34	for	A+B
5.75	-	6.25	for	A-B
2.37	-	2.63	for	B+C
1.40	-	1.60	for	B-C

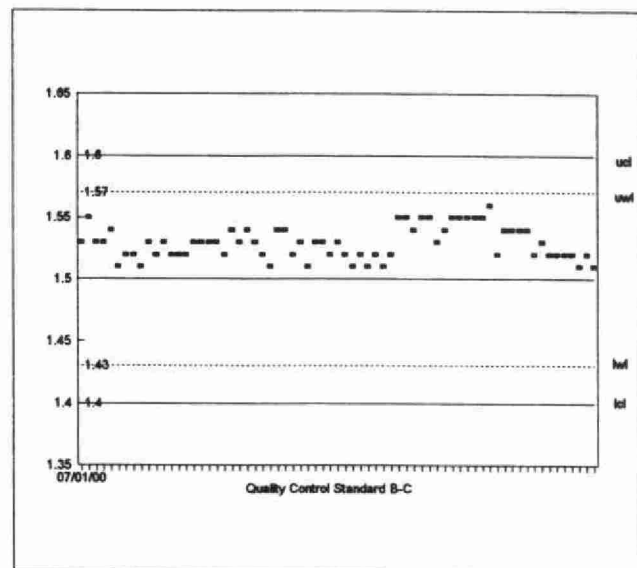
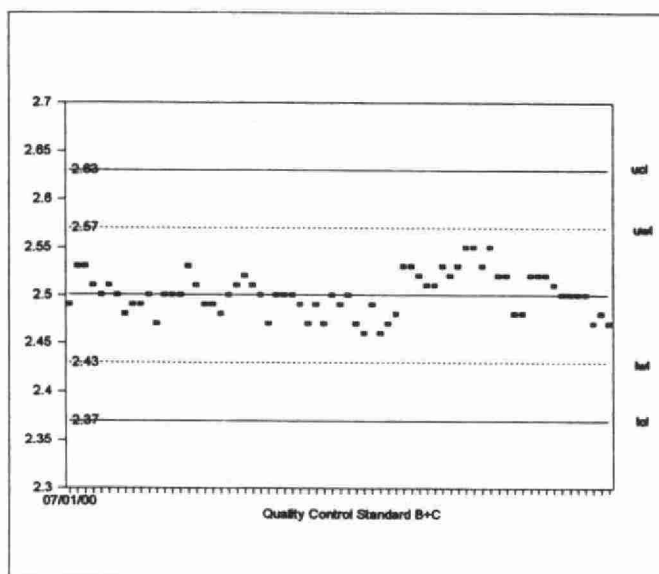
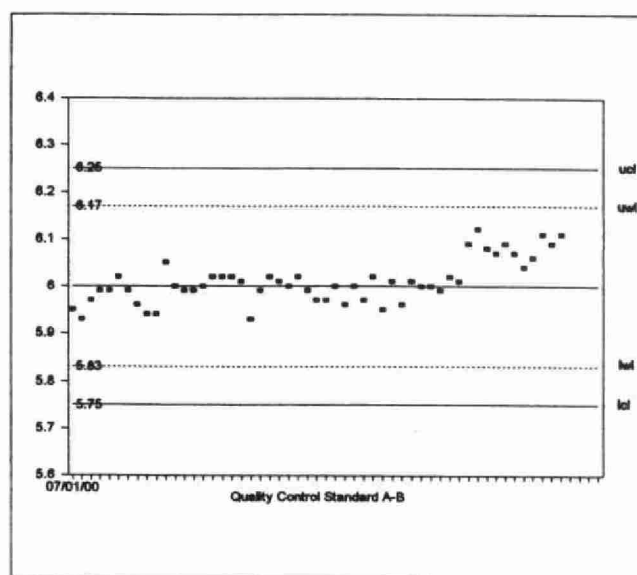
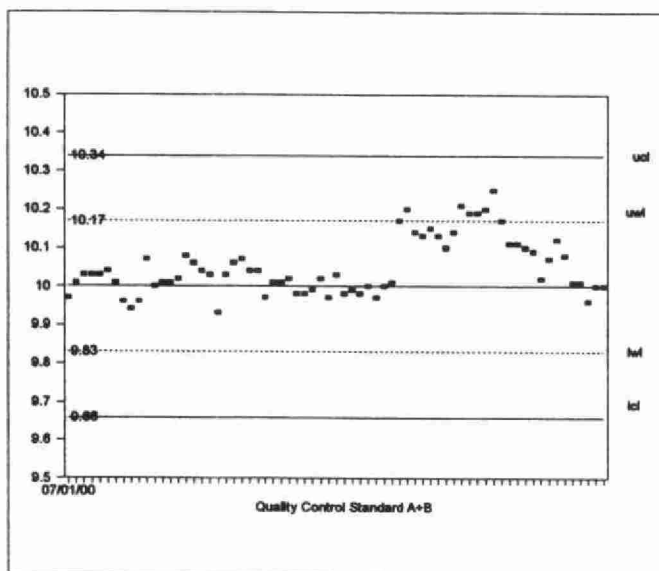
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
83	0.00 - 1.00	0.0610	13.3
43	1.01 - 2.00	0.0244	1.7
55	2.01 - 5.00	0.0180	0.5
8	5.01 -10.00	0.0509	0.8
189	Overall	0.0444	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	69	-0.0294	0.0084

SILICON, REACTIVE SILICATES (mg/L as Si)
QUALITY CONTROL DATA FROM 07/01/00 TO 18/12/00
E3370



SOLIDS, DISSOLVED

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	Before '61
Method Reference No.	E3188	Reporting Unit	mg/L
LIMS Product Code	TSD3188,DS3188,DIGN3188	Supervisor	P. Wilson
Sample Type/Matrix	Sludge, Effluent, Raw Sewage, Industrial Waste, Process Water, Surface Water, Drinking Water, Ground Water, Leachate		

SAMPLING:

Quantity Required	125 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Sample is filtered under moderate suction through a Whatman 934AH grade glass fibre filter. Generally 100 mL of filtrate (alternate 50 mL) is pipetted into a preweighed Teflon dish, dried at $103 \pm 2^\circ\text{C}$, and stored in a desiccator for at least 24 hours. The dissolved solids content is calculated by subtracting the original dish mass from the dried residue + dish mass. Data collection, calculations, and transfer of results to LIMS are controlled by a microcomputer system.

INSTRUMENTATION:

Balance (5 decimal places), drying oven, suction filtration apparatus, dishes (Teflon).
Microcomputer system with appropriate software.

REPORTING:

Maximum Significant Figures: 3	Current W value: 2	Current T value: 10
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CALIBRATION:

Balance zero
Balance internal calibration is performed daily.

CONTROLS:

Calibration	2 S class weights, e.g. QCA (results in grams)
Drift	Balance is reset to zero after every 10 weighings by the microcomputer.
Recovery	2 standards, e.g. R1
Method Blank	100 mL Pure Water.

SOLIDS, DISSOLVED (E3188)
(mg/L)

QUALITY CONTROL DATA FROM 07/01/00 TO 19/12/00

CALIBRATION CONTROL:

	n	Expected Mass (g)	Mean Mass Measured (g)	Mean Bias (g)	Standard Deviation (1)
A:	80	50.00	50.0006	0.0006	0.00005
B:	80	30.00	30.0003	0.0003	0.00006
A+B:		80.00	80.0009	0.0009	0.00009
A-B:		20.00	20.0003	0.0003	0.00006

s.d.(AB)

S(between runs): 0.00005

Sw(within run): 0.00004

S/Sw: 1.2

The calibration is accepted if the calibration control values (mean mass measured) obtained lie within the ranges expressed in grams:

80.0005 - 80.0013 for A+B
20.0000 - 20.0006 for A-B

RECOVERIES:

Number of Data	Expected Concentration (mg/L)	Mean Concentration Measured (mg/L)	Standard Deviation (1)
79	2000.0	1986.58	9.5
78	500.0	489.72	7.7

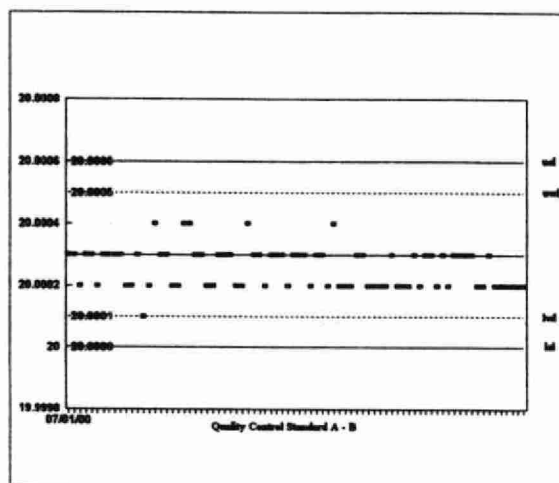
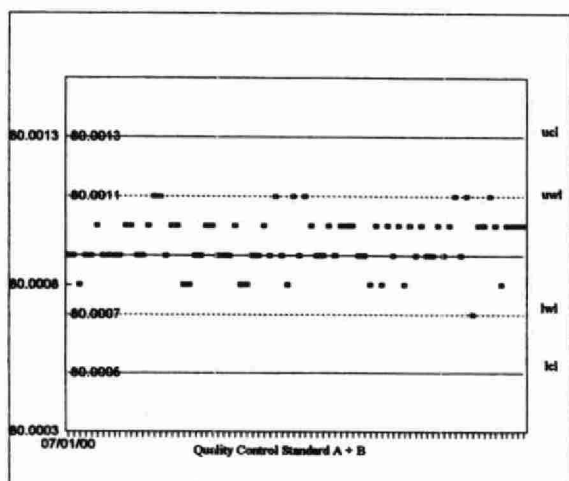
DUPLICATES:

n Data Pairs	Sample Concentration Span (mg/L)	Standard Deviation (2)	Coefficient of variation(%)
43	0 - 500	8.2835	2.7
101	501 - 1000	9.8267	1.4
38	1001 - 5000	13.1822	0.8
182	Overall	10.2996	

OTHER CHECKS:

	n	Data Mean (mg/L)	Standard Deviation (1)
Blank	79	-5.9958	8.4292

DISSOLVED, SOLIDS (mg/L)
QUALITY CONTROL DATA FROM 07/01/00 TO 19/12/00
E3188



SOLIDS, SUSPENDED

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	Before '81
Method Reference No.	E3188	Reporting Unit	mg/L
LIMS Product Code	TSD3188, SS3188	Supervisor	P. Wilson
Sample Type/Matrix	Sludge, Effluent, Raw Sewage, Industrial Waste, Process Water, Surface Water, Drinking Water, Ground Water, Leachate		

SAMPLING:

Quantity Required	5-500 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

An appropriately shaken sample volume (5 to 500 mL) is pipetted or quickly poured into a graduated cylinder, and the volume is measured. The aliquot is then filtered under moderate suction through a preweighed Whatman 934AH glass fibre filter. The graduated cylinder and then the filter are washed with a total of 50 mL distilled water. The filter is dried at 103-105°C, and suspended solids content is calculated by subtracting the original filter mass from the dried filter mass. Data collection, calculations, and transfer of results to LIMS are controlled by a microcomputer system.

INSTRUMENTATION:

Balance (5-decimal places), drying oven, suction filtration apparatus.
Microcomputer system with appropriate software.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.5	Current T value: 2.5
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CONTROLS:

Calibration	2 S class weights, e.g. QCA (results in grams)
Drift	Balance is reset to zero after every 10 weighings by the microcomputer.
Recovery	2 standards, e.g. R1
Method Blank	Filter washed with 500 mL distilled water

NOTES:

A standard correction factor (-0.00022g) was applied to all filters to account for weight loss during filtering. A new set of Q.C. weights was introduced for the year 2000, along with new limits for the weights.

SOLIDS, SUSPENDED (E3188)

(mg/L)

QUALITY CONTROL DATA FROM 20/01/00 TO 22/12/00

Laboratory Unit: Solids

CALIBRATION CONTROL: (QC data from SS3188)

	n	Expected Mass (g)	Mean Mass Measured (g)	Mean Bias (g)	Standard Deviation (1)
C:	266	0.50	0.4999	-0.0001	0.00001
D:	266	0.05	0.0499	-0.0001	0.00004
C+D:		0.55	0.5499	-0.0001	0.00004
C-D:		0.45	0.4500	0.00003	0.00005

s.d.(CD) S(between runs): 0.00003

Sw(within run): 0.00003

S/Sw: 1.0

The calibration is accepted if the calibration control values (mean mass measured) obtained within the ranges expressed in grams:

0.54964 - 0.55005 for C+D
0.44984 - 0.45001 for C-D

RECOVERIES:

Number of Data	Expected Concentration (mg/L)	Mean Concentration Measured (mg/L)	Standard Deviation (1)
265	200.0	194.04	2.3019
265	50.0	49.25	1.2639

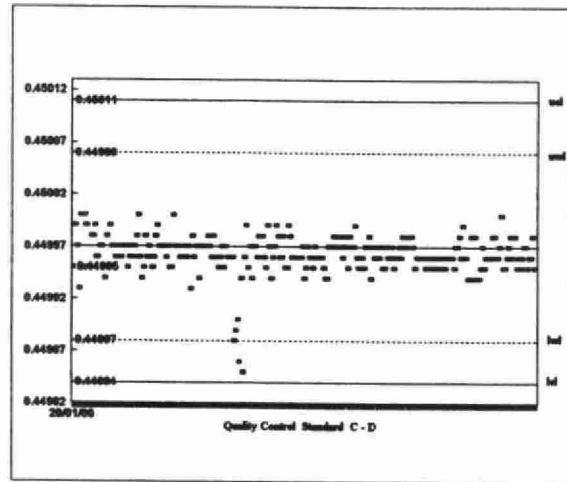
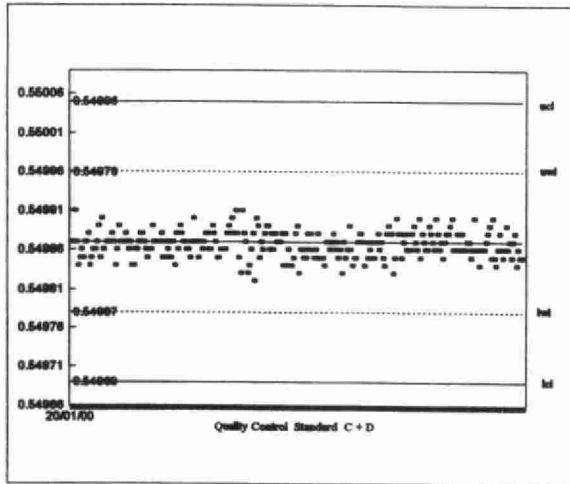
DUPLICATES:

n Data Pairs	Sample Concentration Span (mg/L)	Standard Deviation (2)	Coefficient of variation(%)
178	0 - 5	0.2939	11.9
84	5.1 - 10.0	0.6066	8.9
112	10.1 - 25	0.9751	6.4
134	25.1 - 100	3.0348	5.9
91	100.1 - 500	10.6612	4.9
14	500.1 - 1000	10.7296	1.6
13	1000.1 - 10000	49.3103	2.1
626	Overall	8.4739	

OTHER CHECKS:

	n	Data Mean (mg/L)	Standard Deviation (1)
Blank	264	0.1741	0.2281

SOLIDS, SUSPENDED (mg/L)
QUALITY CONTROL DATA FROM 20/01/00 TO 22/12/00
E3188



SOLIDS, SUSPENDED IGNITED
(Particulate Ash and Particulate Loss On Ignition)

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	Before '61
Method Reference No.	E3188	Reporting Unit	mg/L
LIMS Product Code	SIGN3188	Supervisor	P. Wilson
Sample Type/Matrix	Sludge, Effluent, Raw Sewage, Industrial Waste, Process Water		

SAMPLING:

Quantity Required	5-500 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

The procedure for particulate solids (SS3188) is followed and the dried residue is ignited at $600 \pm 50^\circ\text{C}$ for one hour in a muffle furnace. The dish is transferred to a desiccator to cool. The particulate ash (fixed solids) is the difference between the final ignited mass plus filter and the original tare weight of the filter, divided by the original sample volume (mL) used for SS3188. The particulate loss on ignition (estimate of volatile suspended solids) is the difference between the final ignited mass plus filter and the residue (suspended solids) plus filter, divided by the original sample volume (mL). Data collection, calculations, and transfer of results to LIMS are controlled by a microcomputer system.

INSTRUMENTATION:

Balance (5 decimal places), muffle furnace, filters, Petri dishes
 Microcomputer system with appropriate software

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.5	Current T value: 2.5
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CONTROLS:

Calibration	2 S class weights, e.g. QCA (results in grams)
Drift	Balance is reset to zero after every 10 weighings by the microcomputer.

SOLIDS, SUSPENDED IGNITED
(Particulate Ash and Particulate Loss On Ignition)

QUALITY CONTROL DATA FROM 17/01/00 TO 22/12/00

CALIBRATION CONTROL:

	n	Expected Mass (g)	Mean Mass Measured (g)	Mean Bias (g)	Standard Deviation (1)
C:	27	0.50	0.4999	-0.00008	0.00001
D:	27	0.05	0.0500	0.00005	0.00001
C+D:		0.55	0.5499	-0.00013	0.00002
C-D:		0.45	0.4500	0.00003	0.00001

s.d.(CD) S(between runs): 0.000011 Sw(within run): 0.000009 S/Sw: 1.3

The calibration is accepted if the calibration control values (mean mass measured) obtained lie within the ranges expressed in grams:

0.54964 - 0.55005 for C+D
0.44984 - 0.45001 for C-D

SOLIDS, SUSPENDED IGNITED (PARTICULATE ASH)

DUPLICATES:

n Data Pairs	Sample Concentration Span (mg/L)	Standard Deviation (2)	Coefficient of variation(%)
46	0 - 100.0	3.3508	15.8
4	100.1 - 500.0	9.9862	4.9
2	500.1 - 1000.0	N.A.	N.A.
16	1000.1 - 5000.0	29.4040	1.4
68	Overall	14.8685	

OTHER CHECKS:

	n	Data Mean (mg/L)	Standard Deviation (1)
Blank	27	-0.0504	0.2257

SOLIDS, SUSPENDED IGNITED
(Particulate Ash and Particulate Loss On Ignition)

SOLIDS, SUSPENDED IGNITED (PARTICULATE LOSS ON IGNITION)

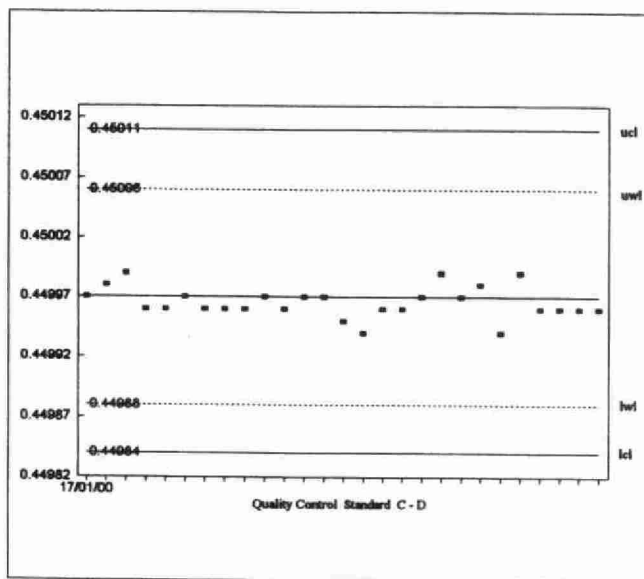
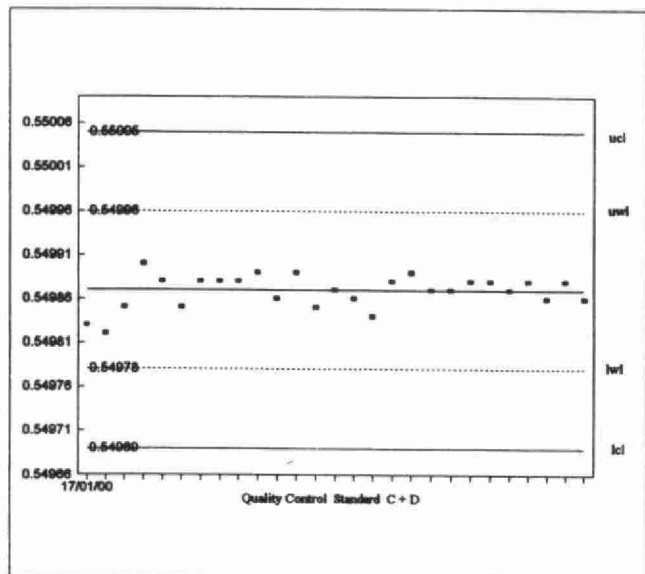
DUPLICATES:

n Data Pairs	Sample Concentration Span (mg/L)	Standard Deviation (2)	Coefficient of variation(%)
32	0 - 50.0	1.7932	6.7
10	50.1 - 100.0	3.7659	4.6
7	100.1 - 1100.0	4.2547	1.6
15	1000.1 - 5000.0	46.6612	1.6
64	Overall	22.7136	

OTHER CHECKS:

	n	Data Mean (mg/L)	Standard Deviation (1)
Blank	25	0.1288	0.1496

Solids, Suspended Ignited
(Particulate Ash and Particulate Loss on Ignition)
 QUALITY CONTROL DATA FROM 17/01/00 TO 22/12/00
 E3188



SOLIDS, TOTAL

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	Before '81
Method Reference No.	E3188	Reporting Unit	mg/L or mg/Kg
LIMS Product Code	TS3188	Supervisor	P. Wilson
Sample Type/Matrix	Sludge, Effluent, Raw Sewage, Industrial Waste, Process Water, Surface Water, Drinking Water, Ground Water, Leachate		

SAMPLING:

Quantity Required	125 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Generally, 100 mL aliquot of sample (alternate 50 mL) is pipetted into a preweighed Teflon dish, dried at 103-105°C, and stored in a desiccator for at least 24 hours. The total residue or solids content is calculated by subtracting the original dish mass from the dried dish mass. Data collection, calculations, and transfer of results to LIMS are controlled by a microcomputer system.

INSTRUMENTATION:

Balance (5 decimal places), drying oven, dishes (Teflon).
Microcomputer system with appropriate software.

REPORTING:

Maximum Significant Figures: 3	Current W value: 2.0	Current T value: 10
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CALIBRATION:

Balance zero
Balance internal calibration performed daily.

CONTROLS:

Calibration	2 S class weights, e.g. QCA (results in grams)
Drift	Balance is reset to zero after every 10 weighings by the microcomputer.
Recovery	2 standards, e.g. R1

SOLIDS, TOTAL (E3188)
(mg/L)
QUALITY CONTROL DATA FROM 17/01/00 TO 27/12/00

CALIBRATION CONTROL: (QC data from TS3188)

	n	Expected Mass (g)	Mean Mass Measured (g)	Mean Bias (g)	Standard Deviation (1)
A:	28	50.00	50.0006	0.0006	0.00007
B:	28	30.00	30.0003	0.0003	0.00007
A+B:		80.00	80.0009	0.0009	0.00013
A-B:		20.00	20.0003	0.0003	0.00005

s.d.(AB) S(between runs): 0.00007 Sw(within run): 0.00004 S/Sw: 1.98

The calibration is accepted if the calibration control values (mean mass measured) obtained lie within the range expressed in grams:

80.0005 - 80.0013 for A+B
20.0000 - 20.0006 for A-B

RECOVERIES:

Number of Data	Expected Concentration (mg/L)	Mean Concentration Measured (mg/L)	Standard Deviation (1)
20	20000.0	20020.4	77.2
20	2000.0	1986.7	10.9

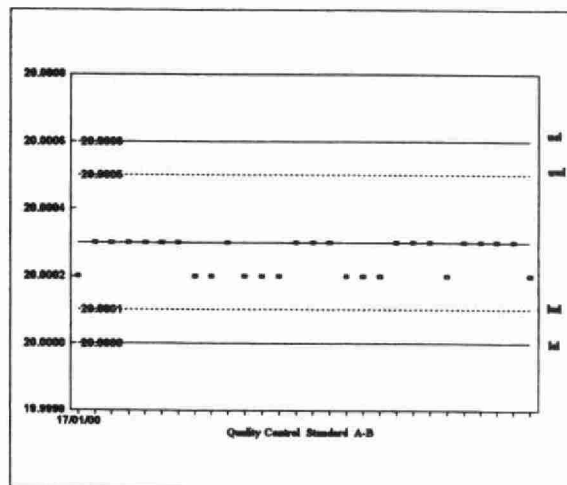
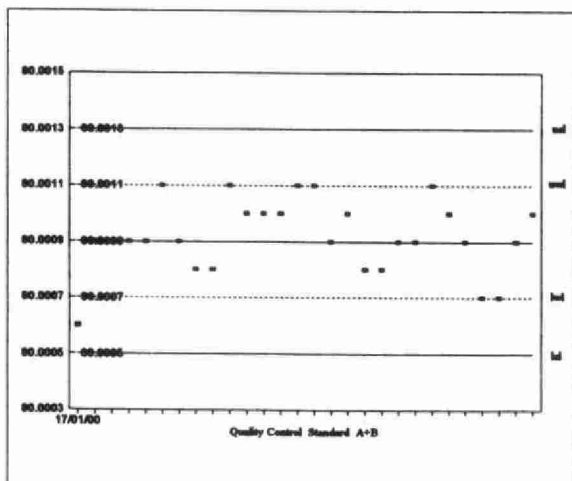
DUPLICATES:

n Data Pairs	Sample Concentration Span (mg/L)	Standard Deviation (2)	Coefficient of variation(%)
9	0 - 6000	14.2931	1.2
10	6001 - 25000	276.7685	1.4
22	25001 - 50000	487.5945	1.4
41	Overall	382.4922	

OTHER CHECKS:

	n	Data Mean (mg/L)	Standard Deviation (1)
Blank	28	-17.8157	10.0321

SOLIDS, TOTAL (mg/L)
Quality Control Data From 17/01/00 To 27/12/00
E3188



SOLIDS, TOTAL IGNITED
(Ash and Loss On Ignition)

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	Before '61
Method Reference No.	E3188	Reporting Unit	mg/L
LIMS Product Code	TIGN3188	Supervisor	P. Wilson
Sample Type/Matrix	Sludge, Effluent, Raw Sewage, Industrial Waste, Process Water		

SAMPLING:

Quantity Required	5-500 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

The procedure for total solids (TS3188) is followed and the dried residue is ignited at $600 \pm 50^{\circ}\text{C}$ for one hour in a muffle furnace. The dish is transferred to a desiccator to cool. The ash (fixed solids) is the difference between the final ignited mass plus filter and the original tare weight of the filter, divided by the original sample volume (mL) used for TS3188. The loss on ignition (estimate of volatile total solids) is the difference between the final ignited mass plus filter and the residue (total solids) plus filter, divided by the original sample volume (mL). Data collection, calculations, and transfer of results to LIMS are controlled by a microcomputer system.

INSTRUMENTATION:

Balance (5 decimal places), muffle furnace, filters, Petri dishes.
Microcomputer system with appropriate software.

REPORTING:

Maximum Significant Figures: 3	Current W value: 2.0	Current T value: 10
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CONTROLS:

Calibration	2 S class weights, e.g. QCA (results in grams)
Drift	Balance is reset to zero after every 10 weighings by the microcomputer.

SOLIDS, TOTAL IGNITED (E3188)
(Ash and Loss On Ignition)
(mg/L)
QUALITY CONTROL DATA FROM 17/01/00 TO 22/12/00

CALIBRATION CONTROL:

	n	Expected Mass (g)	Mean Mass Measured (g)	Mean Bias (g)	Standard Deviation (1)
A:	15	50.00	50.0006	0.0006	0.00006
B:	15	30.00	30.0003	0.0003	0.00008
A+B:		80.00	80.0009	0.0009	0.00014
A-B:		20.00	20.0003	0.0003	0.00004

s.d.(AB) S(between runs): 0.00007 Sw(within run): 0.00004 S/Sw: 2.5

The calibration is accepted if the calibration control values (mean mass measured) obtained lie within the ranges expressed in grams:

80.0005 - 80.0013 for A+B
20.0000 - 20.0006 for A-B

SOLIDS, TOTAL IGNITED (DRY)

RECOVERIES:

Number of Data	Expected Concentration (mg/L)	Mean Concentration Measured (mg/L)	Standard Deviation (1)
7	20000.0	20012.1	72.70
7	2000.0	1988.7	12.04

DUPLICATES:

n Data Pairs	Sample Concentration Span (mg/L)	Standard Deviation (2)	Coefficient of variation(%)
0	0 - 5000	N.A.	N.A.
0	5001 - 15000	N.A.	N.A.
3	15001 - 25000	361.2	1.5
15	25001 - 50000	476.2	1.3
18	Overall	459	

**SOLIDS, TOTAL IGNITED cont'd
(Ash and Loss On Ignition)**

SOLIDS, TOTAL IGNITED (ASH)

DUPLICATES:

n Data Pairs	Sample Concentration Span (mg/L)	Standard Deviation (2)	Coefficient of variation(%))
0	0 - 3000	N.A.	N.A.
3	3001 - 10000	141.5	1.6
15	10001 - 15000	109.6	1.0
18	150001 - 25000	N.A.	N.A.
	Overall	115.5	

OTHER CHECKS:

Ashed	n	Data Mean (mg/L)	Standard Deviation (1)
Blank	7	-23.6429	4.7286

SOLIDS, TOTAL IGNITED (LOSS ON IGNITION)

DUPLICATES:

n Data Pairs	Sample Concentration Span (mg/L)	Standard Deviation (2)	Coefficient of variation(%)
6	0 - 5000	167.7	1.2
4	5001 - 15000	420.0	2.0
7	15001 - 25000	493.0	1.6
17	Overall	389.3	

OTHER CHECKS:

LOI	n	Data Mean (mg/L)	Standard Deviation (1)
Blank	7	-1.4000	5.2751

SULPHATE

IDENTIFICATION:

Laboratory Unit	Water Chemistry	Method Introduced	01/04/78
Method Reference No.	E3004	Units	$\mu\text{g}/\text{m}^3$ as SO_4
LIMS Product Code	ANION3004	Supervisor	P. Wilson
Sample Type/Matrix	Air, HiVol Glass Fibre, Quartz and Polyflon, Other Filters and Puff		

SAMPLING:

Quantity Required	3/4" or 1.9cm strip from 8"x10" filter
Container	50 mL polypropylene tube

SAMPLING PREPARATION:

A 3/4" strip is cut in pieces and deposited into a 50 mL polypropylene tube. 50 mL of Pure-Water is added to the tube. The tube is placed on a horizontal shaker for approximately 1 hour. The supernatant is then filtered into a 15 mL plastic tube and analysed.

ANALYTICAL PROCEDURE:

Sulphate separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of sodium bicarbonate and sodium carbonate and a conductivity detector. The concentration of sulphate (mg/L) is determined by the comparison of the analyte peak area count to that of a series of standards. The analyte result is corrected for the filter blank before the final calculation is made. The result is reported as $\mu\text{g}/\text{m}^3$ as SO_4 .

Chloride and nitrate are determined simultaneously.

INSTRUMENTATION:

Horizontal Shaker, ion chromatographic system plus a PC with ChromPerfect software and DT2804 card for automated sample injection, timing, and data processing.

REPORTING:

Maximum Significant Figures: 2	Current W value: $0.1 \mu\text{g}/\text{m}^3$	Current T value: $0.5 \mu\text{g}/\text{m}^3$
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CALIBRATION:

6 standards

CONTROLS:

Calibration	MB, IS(n), CS1, and CS2
Drift	Duplicate plus 2 standards approximately every 20 samples

SULPHATE cont'd

NOTES:

To convert unit from mg/L to $\mu\text{g}/\text{m}^3$, the final concentration of SO_4 in mg/L is multiplied by the following formula:

$$\text{Result (mg/L)} \times 50\text{mL} \times (63/6.75) / \text{air volume} = \mu\text{g}/\text{m}^3$$

Where: 63 is the area of the filter exposed and 6.75 is the sample aliquot area in square inch.

SULFATE (E3004)

QUALITY CONTROL DATA FOR 2000

Analytical Range: to 28.61 $\mu\text{g}/\text{m}^3$

DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
37	0 - 2.9	0.053	3.8
25	3.0 - 7.1	0.083	1.8
12	7.2 - 14.3	0.110	1.0
7	14.4 - 24.9	0.167	0.9
81	Overall	0.087	

SULPHATE

IDENTIFICATION:

Laboratory Unit	Water Chemistry	Method Introduced	01/01/86
Method Reference No.	E3013	Units	µg/g as SO ₄
LIMS Product Code	ANION3013, SUL3013	Supervisor	P. Wilson
Sample Type/Matrix	Soil and Sediment		

SAMPLING:

Quantity Required	20 g
Container	glass or plastic

SAMPLING PREPARATION:

A 3.0 g sample air dried, sieved soil or air dried sieved and ground sediment is placed in a 50 mL centrifuge tube and shaken with 30 mL Pure-DW for 1 hour on a shaker. Samples are centrifuged, membrane filtered and analyzed for chloride and sulphate by ion chromatography.

ANALYTICAL PROCEDURE:

Sulphate separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of sodium bicarbonate and sodium carbonate and a conductivity detector. The concentration of sulphate (mg/L) is determined by the comparison of the analyte peak area count to that of a series of standards. The result is reported as µg/g as SO₄.

Chloride is determined simultaneously.

INSTRUMENTATION:

Horizontal Shaker, ion chromatographic system plus a PC with ChromPerfect software and DT2804 card for automated sample injection, timing, and data processing.

REPORTING:

Maximum Significant Figures: 2	Current W value: 0.5 µg/g	Current T value: 2.5 µg/g
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CALIBRATION:

8 standards

CONTROLS:

Calibration	MB, IS(n), CS1, and CS2
Drift	Duplicate plus 2 standards approximately every 20 samples

SULFATE (E3013)

QUALITY CONTROL DATA FOR 2000

Analytical Range: to 1000 $\mu\text{g/g}$

DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
83	0 - 200	1.4935	4.4
7	201 - 500	4.4053	1.5
4	501 - 1000	7.3410	1.2
94	Overall	2.3891	

SULPHATE

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	01/04/82
Method Reference No.	E3172	Reporting Unit	mg/L as SO ₄
LIMS Product Code	SULP3172	Supervisor	P. Wilson
Sample Type/Matrix	Drinking Water, Surface Water, Ground Water, Leachates, Effluent, Industrial Waste, Raw Sewage		

SAMPLING:

Quantity Required	50 mL
Container	Glass or plastic

ANALYTICAL PROCEDURE:

Sulphate is separated from other anions in the samples by automated suppressed ion chromatography using an eluent mixture of 0.003 M sodium bicarbonate and 0.0024 M sodium carbonate with conductivity detection. The concentration of sulphate in mg/L as SO₄ is determined by comparison of the sample scan to a series of standard scans.

INSTRUMENTATION:

Basic modular continuous flow ion chromatographic system plus control module (in-house design) for automated sample introduction and timing.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.5	Current T value: 2.5
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CALIBRATION:

BL plus 10 standards

CONTROLS:

Calibration	LTBL plus 2 standards, e.g. QCA
Drift	1 standard every 10 samples

SULPHATE (E3172)

QUALITY CONTROL DATA FROM 11/01/00 TO 28/12/00

Full Scale: to 100.0 mg/L as SO₄

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Mean Bias	Standard Deviation (1)
A:	57	80.0	81.3	1.3	0.6253
B:	57	20.0	20.2	0.2	0.4549
A+B:	57	100.0	101.5	1.5	0.8585
A-B:	57	60.0	61.1	1.1	0.6774

s.d.(AB) S(between runs): 0.53 Sw(within run): 0.46 S/Sw: 1.16

The calibration is accepted if the calibration control values obtained lie within the ranges:

97.2 - 102.8 for A+B
57.9 - 62.1 for A-B

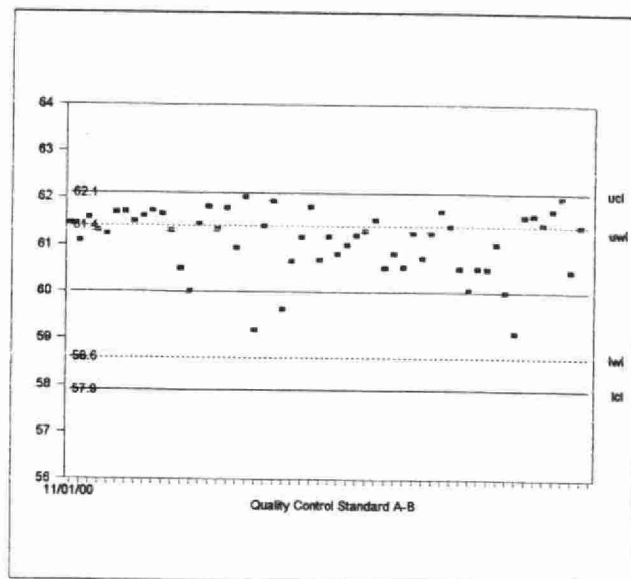
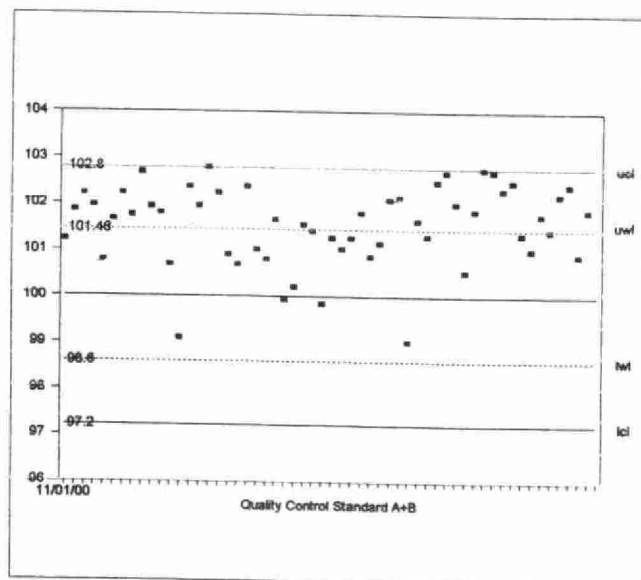
DUPLICATES:

n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
35	0.0 - 10.0	0.3492	7.0
33	10.1 - 20.0	0.8094	5.5
60	20.1 - 50.0	0.9065	3.1
24	50.1 - 100.0	1.4247	1.9
152	Overall	0.9029	

OTHER CHECKS:

	n	Mean	Standard Deviation (1)
Long Term Blank	57	-0.0802	0.4999

SULPHATE (mg/L as SO₄)
QUALITY CONTROL FROM 11/01/00 TO 28/12/00
E3172



TURBIDITY

IDENTIFICATION:

Laboratory	Water Chemistry	Method Introduced	Before '74
Method Reference No.	E3311	Reporting Unit	FTU
LIMS Product Code	TURB3311	Supervisor	P. Wilson
Sample Type/Matrix	Surface Water, Ground Water, Effluent, Drinking Water, Industrial Waste, Process Water, Leachate		

SAMPLING:

Quantity Required:	50 mL
Container:	Glass or plastic

ANALYTICAL PROCEDURE:

The instrument is standardized with sealed standards which are prepared commercially and rated in Formazin Turbidity Units. Samples are placed in the turbidimeter, and results in FTU are read directly from the digital output. Turbidity measurement are based on light scattering at 90 plus or minus 30 degrees of rotation. The instrument compensates for sample colour.

INSTRUMENTATION:

-Hach Ratio/XR Model Turbidimeter modified to accept control signals from robot controller, electronic interphase, Zymark ZYMATE 11 Laboratory Robot System, IBM PC computer.

REPORTING:

Maximum Significant Figures: 3	Current W value: 0.05	Current T value: 0.25
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CALIBRATION:

BL plus formazin standards (once every four months)

CONTROLS:

Calibration:	5 standards, e.g. QCA
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TURBIDITY (E3311)

QUALITY CONTROL DATA FROM 06/01/00 TO 21/12/00

Full Scale: to 2000 FTU

CALIBRATION CONTROL:

	n	Expected Concentration	Mean Concentration	Standard Deviation (1)
A:	165	2.0	1.59	0.0288
B:	165	20.0	15.65	0.2690
C:	165	200.0	152.80	1.9794
D:	165	2000.0	1363.60	37.2591

On any given day the calibration is accepted if the values obtained lie within the ranges:

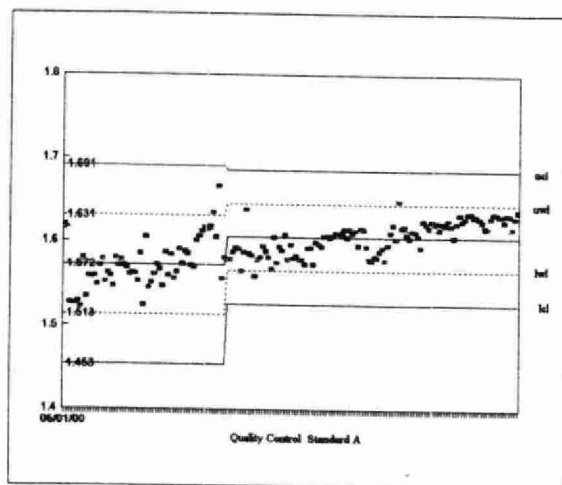
1.453	-	1.691	for	A (Jan-Jun)
1.525	-	1.685		A (Jul-Dec)
14.525	-	16.186	for	B (Jan-Jun)
15.322	-	16.302		B (Jul-Dec)
145.9	-	155.1	for	C (Jan-Jun)
149.5	-	158.5		C (Jul-Dec)
1369	-	1451	for	D (Jan-Jun)
1293	-	1379	for	D (Jul-Dec)

	n	Data Mean	Standard Deviation (1)
Stray Light	165	0.0315	0.0031

DUPLICATES:

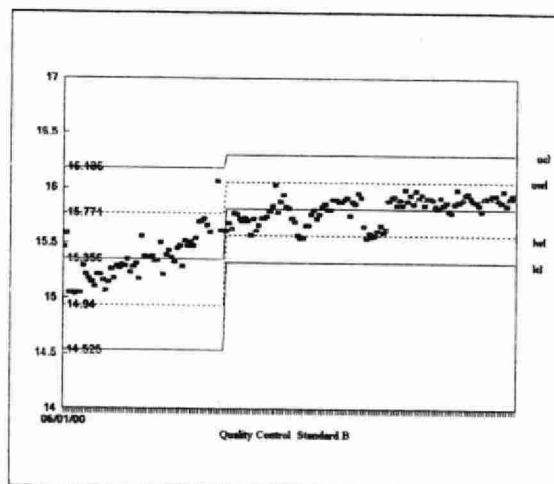
n Data Pairs	Sample Concentration Span	Standard Deviation (2)	Coefficient of variation(%)
304	0.0 - 2.0	0.0375	7.6
118	2.1 - 20.0	0.3255	4.8
31	21.0 - 200	2.2289	5.0
2	201 - 2000	N.A.	N.A.
455	Overall	1.3716	

Turbidity
Quality Control Data from 06/01/00 to 21/12/00
E3311



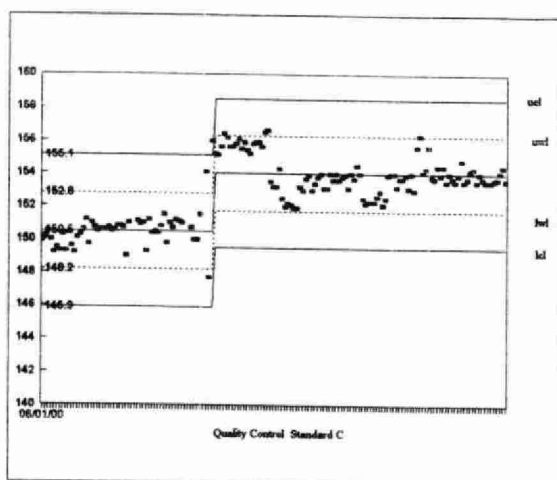
Jan-June

July-Dec



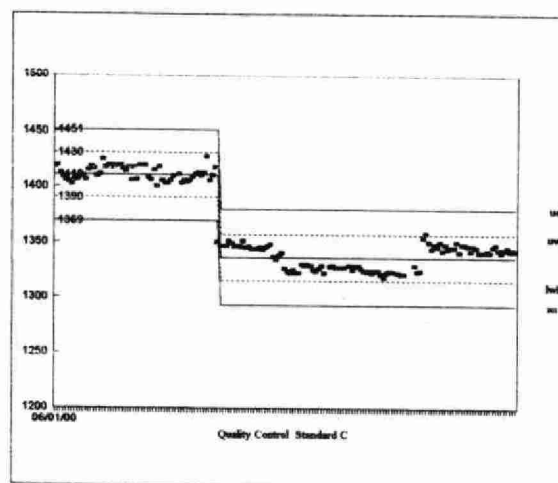
Jan-June

July-Dec



Jan-June

July-Dec



Jan-June

July-Dec

PART 3.0
MICROBIOLOGY

3.1 Quality Control Program, Microbiology Unit

Performance Criteria

Analyses of samples in the Microbiology Unit are performed using approved methodologies, by trained technologists. Safety measures have been incorporated into the methodologies to ensure that all analytical procedures are functioning properly, minimizing the potential to identify and report false positive or negative results. This report focuses on the quality control implemented during sample analyses. Information regarding the implementation of quality control procedures for sample containers, monitoring of the Pure Water supply, media preparation and storage, equipment monitoring are described by the Laboratory Services Branch (4) and Microbiology Unit Standard Operating Procedures (SOPs), approved Microbiology Methods and Lab Services Branch Quality Assurance Manual (2).

Membrane Filtration

Blank Control Analyses

A control (sterile buffered dilution water) sample is processed between each sample analyzed. The control sample is processed in a manner similar to the regular sample including volume, agar used, incubation time and temperature. The blank control should remain free of any bacterial growth.

Duplicate Analyses

Approximately five percent of the samples are analyzed in duplicate per day. The data are accumulated for each parameter and a "within-run" standard deviation is calculated to give a measure of the repeatability of the results.

Presence-Absence Procedure

Blank Control Analyses

Approximately five percent of samples analyzed per day include, a blank control sample prepared by adding a 99 mL dilution blank (sterile, buffered dilution water) to P-A broth and incubating it along with the regular P-A bottles. The blank control should remain free of any bacterial growth and there should be no change in the colour of the broth. Identification of growth or colour change in the control blank requires follow-up of sterility checks in both the P-A broth and the dilution blanks.

Heterotrophic Spread Plate

Blank Control Analyses

Approximately five percent of samples analyzed per day include inoculating a Plate Count agar plate with 0.1 mL of sterile buffered dilution water and incubating it along with the regular Plate Count agar plates ($35\pm0.5^{\circ}\text{C}$, 48 ± 3 hours).

Duplicate Analyses

Approximately five percent of samples are analyzed in duplicate per day. The data are accumulated for each parameter and a "within-run" standard deviation is calculated to give a measure of the repeatability of the results.

Blank Analyses Corrective Action

The presence of bacterial growth on any control sample by the above techniques (Membrane Filtration, PA Broth, Heterotrophic Spread Plate) indicates inaccurate technique. The supervisor must be consulted with regards to determining follow-up and corrective action. Reporting of results may be tempered by the presence of bacterial growth on these control samples and data qualifying remarks codes would be noted on the final report. Records of all control samples are maintained in the laboratory.

3.2 PERFORMANCE SUMMARIES

MICROBIOLOGY

***Escherichia coli* (EC)**

IDENTIFICATION:

Laboratory	Microbiology	Method Introduced	1979
Method Reference No.	E3226	Reporting Unit	Present/Absent per 100 mL
LIMS Product Code	PA3226	Supervisor	R. Schop
Sample Type/Matrix	Drinking Water		

SAMPLING:

Quantity Required:	100 mL
Container:	Plastic, ring sealed
Preservative:	Sodium thiosulphate

ANALYTICAL PROCEDURE:

Sample aliquots are added to P-A broth, incubated ($35\pm0.5^{\circ}\text{C}$, for up to 72 ± 3 hours) and checked for growth, gas and acid production. A presumptive positive is identified as the detection of gas and acid production within 72 hours of incubation. Following the identification of a presumptive positive, confirmatory tests for *Escherichia coli* are conducted according to the method.

INSTRUMENTATION:

Micropipette, sterile micropipette tip, sterile graduated cylinder, bunsen burner, incubators, UV lamp

REPORTING:

Present / Absent per 100 mL

CONTROLS:

Analytical	Negative Control(5% per day) -Sterile buffered dilution water
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NOTES:

*PA3226 is used for the detection of EC and TC. Confirmatory testing using ECMug is required.

***Escherichia coli* (EC)**

E3226
QUALITY CONTROL DATA FOR 2000

Present/Absent per 100 mL

OTHER CHECKS:

	n	number of blanks with growth
Control Blanks	401	6

Escherichia coli (EC)

IDENTIFICATION:

Laboratory	Microbiology	Method Introduced	1979
Method Reference No.	E3371	Reporting Unit	CFU per 100 mL
LIMS Product Code	EC3371, *TCEC3371,*ECFS3371 *,ECFSPS3371	Supervisor	R. Schop
Sample Type/Matrix	Sediment, Sludge, Soil, Effluent, Industrial Waste, Process Water, Raw Sewage, Drinking Water (Raw Water), Ground Water, Leachate, Precipitation, Surface Water		

SAMPLING:

Quantity Required:	100 mL
Container:	Plastic, ring sealed
Preservative:	Sodium thiosulphate

ANALYTICAL PROCEDURE:

Sample aliquots are passed through a 0.45 µm pore size, cellulose filter. The membrane filter is then placed onto MFC-BCIG agar plate and incubated 44.5±0.5°C, 24±2 hours. Target colonies formed on the membrane filter are recorded per 100 mL of sample.

INSTRUMENTATION:

Filtration assembly, sterile membrane filters, sterile graduated cylinders, sterile pipets, bunsen burner, incubators, microscope, Quebec colony counter

REPORTING:

Maximum Significant Figures: 2	Current W value: 0	Current T value: Not Applicable
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CONTROLS:

Analytical	Duplicate samples (5% per day) Blank filter between samples
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NOTES:

* TCEC3371,*ECFS3371*,ECFSPS3371 are mixed parameter product codes. See individual tests TC,FS,PSA, for details on medium used and incubation.

***Escherichia Coli* (EC)**

E3371

QUALITY CONTROL DATA FOR 2000

CFU /100 mL

DUPLICATES:

n Data Pairs	Counts per Filter	Mean Difference	Standard Deviation (2)	Coefficient of Variation (%)
68	0 - 30	2.47	2.43	0.09
17	31 - 75	9.59	9.50	0.01
14	76 - 150	7.00	7.31	0.05

OTHER CHECKS:

	n	number of blanks with growth
Control Blanks	2815	1

***Escherichia coli* (EC)**

IDENTIFICATION:

Laboratory	Microbiology	Method Introduced	1998
Method Reference No.	E3407	Reporting Unit	CFU per 100mL
LIMS Product Code	*TCEC3407	Supervisor	R. Schop
Sample Type/Matrix	Drinking Water		

SAMPLING:

Quantity Required:	100 mL
Container:	Plastic, ring sealed
Preservative:	Sodium thiosulphate

ANALYTICAL PROCEDURE:

Sample aliquots are passed through a 0.45 µm pore size, cellulose filter. The membrane filter is then placed onto DC agar plate and incubated 35±0.5°C, 24±2 hours. Target colonies formed on the membrane filter are recorded per 100 mL of sample.

INSTRUMENTATION:

Filtration assembly, sterile membrane filters, sterile graduated cylinders, bunsen burner, incubator, microscope, Quebec colony counter

REPORTING:

Maximum Significant Figures: 2	Current W value: 0	Current T value: Not Applicable
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CONTROLS:

Analytical	Duplicate samples (5% per day) Blank filter between samples
------------	--

NOTES:

*TCEC3407 is a mixed parameter product code. The same medium and incubation time are used to determine both parameters TC and EC.

***Escherichia Coli* (EC)**

E3407

QUALITY CONTROL DATA FOR 2000

CFU /100 mL

DUPLICATES:

n Data Pairs	Counts per Filter	Mean Difference	Standard Deviation (2)	Coefficient of Variation (%)
15	0 - 30	0.13	0.35	0.1
0	31 - 75	N.A.	N.A.	N.A.
0	76 - 150	N.A.	N.A.	N.A.

OTHER CHECKS:

	n	number of blanks with growth
Control Blanks	8406	0

FAECAL STREPTOCOCCI (FS)

IDENTIFICATION:

Laboratory	Microbiology	Method Introduced	1979
Method Reference No.	E3371	Reporting Unit	CFU per 100 mL
LIMS Product Code	FS3371,*ECFS3371, *ECFSPS3371	Supervisor	R. Schop
Sample Type/Matrix	Sediment, Sludge, Soil, Effluent, Industrial Waste, Process Water, Raw Sewage, Drinking Water (Raw Water), Ground Water, Leachate, Precipitation, Surface Water		

SAMPLING:

Quantity Required:	100 mL
Container:	Plastic, ring sealed
Preservative:	Sodium thiosulphate

ANALYTICAL PROCEDURE:

Sample aliquots are passed through a 0.45 µm pore size, cellulose filter. The membrane filter is then placed onto mEnterococcus agar plate and incubated 35±0.5°C, 48±3 hours. Target colonies formed on the membrane filter are recorded per 100 mL of sample.

INSTRUMENTATION:

Filtration assembly, sterile membrane filters, sterile graduated cylinders, sterile pipets, bunsen burner, incubators, microscope, Quebec colony counter

REPORTING:

Maximum Significant Figures: 2	Current W value: 0	Current T value: Not Applicable
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CONTROLS:

Analytical	Duplicate samples (5% per day) Blank filter between samples
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NOTES:

ECFS3371,ECFSPS3371 are mixed parameter product codes. See individual tests EC,PSA, for details on medium used and incubation.

FAECAL STREPTOCOCCI (FS)

E3371
QUALITY CONTROL DATA FOR 2000

CFU /100 mL

DUPLICATES:

n Data Pairs	Counts per Filter	Mean Difference	Standard Deviation (2)	Coefficient of Variation (%)
49	0 - 30	2.55	2.84	8.04
20	31 - 75	6.25	4.48	20.1
7	76 - 150	10.86	4.49	20.1

OTHER CHECKS:

	n	number of blanks with growth
Control Blanks	2815	1

HETEROTROPHIC PLATE COUNT(HPC)

IDENTIFICATION:

Laboratory	Microbiology	Method Introduced	1998
Method Reference No.	E3408	Reporting Unit	CFU per 1mL
LIMS Product Code	PC3408	Supervisor	R. Schop
Sample Type/Matrix	Drinking Water, Ground Water, Surface Water		

SAMPLING:

Quantity Required:	100 mL
Container:	Plastic, ring sealed
Preservative:	Sodium thiosulphate

ANALYTICAL PROCEDURE:

Sample aliquot is inoculated onto a Plate Count agar plate with a micropipette. The sample is then spread onto the plate using a glass rod and an electronic turntable. The plate is then incubated $35\pm0.5^{\circ}\text{C}$, 48 ± 3 hours and checked for growth. Target colonies formed on the plate are recorded per 1 mL of sample

INSTRUMENTATION:

Micropipette, sterile micropipette tips, sterile glass rod, electronic turntable, incubator, Quebec colony counter

REPORTING:

Maximum Significant Figures: 2	Current W value: 0	Current T value: Not Applicable
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CONTROLS:

Analytical	Duplicates (5% per day) Negative control per run- open air plate Negative control (5% per day) - glass rod check
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HETEROTROPHIC PLATE COUNT (HPC)

E3408

QUALITY CONTROL DATA FOR 2000

CFU /1 mL

DUPLICATES:

n Data Pairs	Counts per Plate	Mean Difference	Standard Deviation (2)	Coefficient of Variation (%)
228	0 - 30	0.31	0.82	0.67
4	31 - 75	7.0	4.90	24.0
1	76 - 150	N.A.	N.A.	N.A.

OTHER CHECKS:

	n	number of blanks with growth
Control Blanks	511	8

INDICATOR ORGANISMS

IDENTIFICATION:

Laboratory	Microbiology	Method Introduced	1979
Method Reference No.	E3226	Reporting Unit	Present/Absent per 100 mL
LIMS Product Code	PA3226	Supervisor	R. Schop
Sample Type/Matrix	Drinking Water		

SAMPLING:

Quantity Required:	100 mL
Container:	Plastic, ring sealed
Preservative:	Sodium thiosulphate

ANALYTICAL PROCEDURE:

Sample aliquots are added to P-A broth and incubated ($35\pm0.5^{\circ}\text{C}$, for up to 72 ± 3 hours) and checked for growth, gas and acid production. A presumptive positive is identified as the detection of gas and acid production within 72 hours of incubation. Following the identification of a presumptive positive, confirmatory tests for indicator organisms are conducted according to the method.

INSTRUMENTATION:

Micropipette, sterile micropipette tip, sterile graduated cylinder, bunsen burner, incubators

REPORTING:

Detected/Not Detected per 100 mL

CONTROLS:

Analytical	Negative Control(5% per day) -Sterile buffered dilution water
------------	---

NOTES:

*PA3226 is used for the detection of indicator organisms. Various media are used in their determinations . See method.

***Pseudomonas aeruginosa* (PSA)**

IDENTIFICATION:

Laboratory	Microbiology	Method Introduced	1979
Method Reference No.	E3371	Reporting Unit	CFU per 100 mL
LIMS Product Code	PSA3371,*ECFSPS3371	Supervisor	R. Schop
Sample Type/Matrix	Sediment, Sludge, Soil, Effluent, Industrial Waste, Process Water, Raw Sewage, Drinking Water (Raw Water), Ground Water, Leachate, Precipitation, Surface Water		

SAMPLING:

Quantity Required:	100 mL
Container:	Plastic, ring sealed
Preservative:	Sodium thiosulphate

ANALYTICAL PROCEDURE:

Sample aliquots are passed through a 0.45 µm pore size, cellulose filter. The membrane filter is then placed onto mPA agar plate and incubated 41.5±0.5°C, 48±3 hours. Target colonies formed on the membrane filter are recorded per 100 mL of sample.

INSTRUMENTATION:

Filtration assembly, sterile membrane filters, sterile graduated cylinders, sterile pipets, bunsen burner, incubators, microscope, Quebec colony counter

REPORTING:

Maximum Significant Figures: 2	Current W value: 0	Current T value: Not Applicable
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CONTROLS:

Analytical	Duplicate samples (5% per day) Blank filter between samples
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NOTES:

*ECFSPS3371 is a mixed parameter product code. See individual test EC, FS for details on medium used and incubation.

***Pseudomonas aeruginosa* (PSA)**

E3371

QUALITY CONTROL DATA FOR 2000

CFU /100 mL

DUPLICATES:

n Data Pairs	Counts per Filter	Mean Difference	Standard Deviation (2)	Coefficient of Variation (%)
84	0 - 30	1.08	1.69	2.9
2	31 - 75	13.0	2.83	8.0
0	76 - 150	N.A.	N.A.	N.A.

OTHER CHECKS:

	n	number of blanks with growth
Control Blanks	2815	1

TOTAL COLIFORM (TC)**IDENTIFICATION:**

Laboratory	Microbiology	Method Introduced	1979
Method Reference No.	E3226	Reporting Unit	Present/Absent per 100 mL
LIMS Product Code	PA3226	Scientist	R. Schop
Sample Type/Matrix	Drinking Water		

SAMPLING:

Quantity Required:	100 mL
Container:	Plastic, ring sealed
Preservative:	Sodium thiosulphate

ANALYTICAL PROCEDURE:

Sample aliquots are added to P-A broth and incubated ($35\pm0.5^{\circ}\text{C}$, for up to 72 ± 3 hours) and checked for growth, gas and acid production. A presumptive positive is identified as the detection of gas and acid production within 72 hours of incubation. Following the identification of a presumptive positive, confirmatory tests for Total Coliforms are conducted according to the method.

INSTRUMENTATION:

Micropipette, sterile micropipette tip, sterile graduated cylinder, bunsen burner, incubators, UV lamp

REPORTING:

Present / Absent per 100 mL

CONTROLS:

Analytical	Negative Control(5% per day) -Sterile buffered dilution water
------------	---

NOTES:

*PA3226 is used for the detection of EC and TC. Confirmatory testing using ECMug is required.

TOTAL COLIFORM (TC)
E3226
QUALITY CONTROL DATA FOR 2000
Present/Absent per 100 mL

OTHER CHECKS:

	n	number of blanks with growth
Control Blanks	401	6

TOTAL COLIFORM (TC)

IDENTIFICATION:

Laboratory	Microbiology	Method Introduced	1979
Method Reference No.	E3371	Reporting Unit	CFU per 100 mL
LIMS Product Code	TC3371, *TCEC3371	Supervisor	R. Schop
Sample Type/Matrix	Sediment, Sludge, Soil, Effluent, Industrial Waste, Process Water, Raw Sewage, Drinking Water (Raw Water), Ground Water, Leachate, Precipitation, Surface Water		

SAMPLING:

Quantity Required:	100 mL
Container:	Plastic, ring sealed
Preservative:	Sodium thiosulphate

ANALYTICAL PROCEDURE:

Sample aliquots are passed through a 0.45 µm pore size, cellulose filter. The membrane filter is then placed onto mEndo LES agar plate and incubated 35±0.5°C, 24±2 hours. Target colonies formed on the membrane filter are recorded per 100 mL of sample.

INSTRUMENTATION:

Filtration assembly, sterile membrane filters, sterile graduated cylinders, sterile pipets, bunsen burner, incubators, microscope, Quebec colony counter.

REPORTING:

Maximum Significant Figures: 2	Current W value: 0	Current T value: Not Applicable
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CONTROLS:

Analytical	Duplicate samples (5% per day) Blank filter between samples
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NOTES:

* TCEC3371 is a mixed parameter product code. See individual test (EC) for details on medium used and incubation.

TOTAL COLIFORM (TC)

E3371

QUALITY CONTROL DATA FOR 2000

CFU /100 mL

DUPLICATES:

n Data Pairs	Counts per Filter	Mean Difference	Standard Deviation (2)	Coefficient of Variation (%)
1	0 - 30	N.A.	N.A.	N.A.
0	31 - 75	N.A.	N.A.	N.A.
0	76 - 150	N.A.	N.A.	N.A.

OTHER CHECKS:

	n	number of blanks with growth
Control Blanks	2815	1

TOTAL COLIFORM (TC)**IDENTIFICATION:**

Laboratory	Microbiology	Method Introduced	1998
Method Reference No.	E3407	Reporting Unit	CFU per 100mL
LIMS Product Code	*TCEC3407	Supervisor	R. Schop
Sample Type/Matrix	Drinking Water		

SAMPLING:

Quantity Required:	100 mL
Container:	Plastic, ring sealed
Preservative:	Sodium thiosulphate

ANALYTICAL PROCEDURE:

Sample aliquots are passed through a 0.45 µm pore size, cellulose filter. The membrane filter is then placed onto DC agar plate and incubated 35±0.5°C, 24±2 hours. Target colonies formed on the membrane filter are recorded per 100 mL of sample.

INSTRUMENTATION:

Filtration assembly, sterile membrane filters, sterile graduated cylinders, bunsen burner, incubator, microscope, Quebec colony counter

REPORTING:

Maximum Significant Figures: 2	Current W value: 0	Current T value: Not Applicable
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CONTROLS:

Analytical	Duplicate samples (5% per day) Blank filter between samples
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NOTES:

*TCEC3407 is a mixed parameter product code. The same medium and incubation time are used to determine both parameters TC and EC.

TOTAL COLIFORM (TC)

E3407

QUALITY CONTROL DATA FOR 2000

CFU /100 mL

DUPLICATES:

n Data Pairs	Counts per Filter	Mean Difference	Standard Deviation (2)	Coefficient of Variation (%)
15	0 - 30	0.33	0.82	0.7
0	31 - 75	N.A.	N.A.	N.A.
0	76 - 150	N.A.	N.A.	N.A.

OTHER CHECKS:

	n	number of blanks with growth
Control Blanks	8046	0

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ABBREVIATIONS

AAll	- Auto Analyzer Model II
AAS	- Atomic Absorption Spectrophotometer
Bl	- Blank
°C	- Degree Centigrade
cm	- Centimetre
CS1	- Check Sample 1
CS2	- Check Sample 2
Date	- Day/Month/Year
DO	- Dissolved Oxygen
EDTA	- Ethylenediaminetetra-Acetic Acid, Disodium Salt, Dihydrate
FTU	- Formazin Turbidity Units
g	- Gram
HZU	- Hazen Units
in ²	- Square Inches
IS(n)	- Internal Standard (n denotes parameter)
kg	- kilogram
L	- Litre
LAB	- Laboratory
LIMS	- Laboratory Information Management System
LTB/L	- Long Term Blank
lcl	- Low Control Limit
lwl	- Low Warning Limit
m ³	- Cubic Metre
M	- Molarity
MB	- Method Blank
meq	- Milliequivalent
mg	- Milligram
min	- Minute
mL	- Millilitre
mm	- Millimetre
N	- Normality
N.A.	- Not Available or Not Applicable
nm	- Nanometre
n	- Number
PC	- Personal Computer
Pure-DW	- Pure Deionized Water

ABBREVIATIONS cont'd

Pure-W	- Pure Water
QC	- Quality Control
QCA	- Quality Control Standard A
QCB	- Quality Control Standard B
QCC	- Quality Control Standard C
QCD	- Quality Control Standard D
R	- Recovery
rpm	- Revolutions Per Minute
RS92	- Reference Standard (in -house)
S	- Between Run Standard Deviation
S ₁	- Standard Deviation (Conventional)
S ₂	- Standard Deviation For Duplicates
S _w	- Standard Deviation Within Run
S. Class	- Weight Classification Designation (not certified)
s.d.	- Standard Deviation
Standard Cal	- Colourimeter setting to control electronic expansion
STD	- Standard
TCU	- True Colour Units
TPTZ	- Ferrous-2,4,6-tri(2'pyridyl)-1,3,5,- triazine
ucl	- Upper Control Limit
uwl	- Upper Warning Limit
μm	- Micrometer
μeq	- Microequivalent
μg	- Microgram
μS	- Micro-Siemen
UV	- Ultra-Violet
V/V	- Concentration based on volume measurements
W40	- Whatman 40 Filters
%	- Percent

Appendix A
W and T values for '00

Parameter	Method Reference No.	Units	Full Scale	W	T
Acidity, Gran	(E3248)	$\mu\text{eq/L H}^+$	1000	5.0	25.0
Acidity, Total Fixed Endpoint	(E3248)	mg/L CaCO_3	1000	0.2	1.0
Alkalinity, Total Fixed Endpoint	(E3218)	mg/L CaCO_3	1000	0.5	2.5
Carbon, Dissolved Inorganic	(E3370)	mg/L C	80.0	0.2	1.0
Carbon, Dissolved Organic	(E3370)	mg/L C	20.0	0.1	0.5
Chloride	(E3004)	$\mu\text{g/m}^3 \text{ Cl}$	14.7	0.1	0.5
Chloride	(E3013)	$\mu\text{g/g Cl}$	-	0.5	2.5
Chloride	(E3016)	mg/L Cl	100	0.2	1.0
Chlorophyll "a"	(E3169)	$\mu\text{g/L}$	-	0.2	1.0
Chlorophyll "a" Acidified	(E3169)	$\mu\text{g/L}$	-	1.0	5.0
Chlorophyll "b"	(E3169)	$\mu\text{g/L}$	-	0.1	0.5
Colour, True	(E3219)	TCU	100	0.2	1.0
Conductivity	(E3218)	$\mu\text{S/cm}$	2000	1	5
Cyanide, Free	(E3015)	mg/L CN^-	0.2	0.001	0.005
		$\mu\text{g/g CN}^-$		0.01	0.05
Cyanide, Total	(E3015)	mg/L CN^-	0.2	0.001	0.005
Cyanide, Total	(E3015)	$\mu\text{g/g CN}^-$		0.01	0.05
Fluoride	(E3369)	mg/L F	2.0	0.01	0.05
Nitrate	(E3004)	$\mu\text{g/m}^3 \text{ NO}_3$	14.7	0.1	0.5
Nitrilotriacetic Acid	(E3406)	mg/L NTA	1.00	0.01	0.05
Nitrogen,					
Ammonia Plus Ammonium	(E3364)	mg/L N	2.0	0.002	0.01
Ammonia Plus Ammonium	(E3366)	mg/L N	50.0	0.05	0.25
Nitrogen, Nitrate Plus Nitrite	(E3364)	mg/L N	5.00	0.005	0.025
Nitrogen, Nitrate Plus Nitrite	(E3366)	mg/L N	50.0	0.05	0.25
Nitrogen, Nitrite	(E3364)	mg/L N	0.200	0.001	0.005
Nitrogen, Nitrite	(E3366)	mg/L N	2.00	0.005	0.025
Nitrogen, Total Kjeldahl	(E3116)	mg/g N	20	0.1	0.5
Nitrogen, Total Kjeldahl	(E3118)	mg/g N	100	0.2	1

Appendix A
W and T values for '00

Parameter	Method Reference No.	Units	Full Scale	W	T
Nitrogen, Total Kjeldahl	(E3367)	mg/L N	2.00	0.02	0.1
Nitrogen, Total Kjeldahl	(E3368)	mg/L N	50.0	0.05	0.25
Oxygen Demand, Biochemical	(E3182)	mg/L O	9.0	0.2	1
Oxygen Demand, Chemical	(E3170)	mg/L O	40.0	1	5
Oxygen Demand, Chemical	(E3246)	mg/L O	500	2	10
pH	(E3218)	-	-	-	-
pH	(E3248)	-	-	-	-
Phenolics, Reactive	(E3179)	µg/L Phenol	50.0	0.2	1.0
Phosphorus,					
Reactive ortho-Phosphate	(E3364)	mg/L P	0.100	0.0005	0.0025
Reactive ortho-Phosphate	(E3366)	mg/L P	10.0	0.02	0.10
Phosphorus, Total	(E3116)	mg/g P	5	0.02	0.10
Phosphorus, Total	(E3118)	mg/g P	8	0.02	0.10
Phosphorus, Total	(E3367)	mg/L P	0.200	0.002	0.01
Phosphorus, Total	(E3368)	mg/L P	10.0	0.02	0.10
Silicon, Reactive Silicates	(E3370)	mg/L Si	10.0	0.02	0.10
Solids, Dissolved	(E3188)	mg/L	-	2	10
Solids, Suspended	(E3188)	mg/L	-	0.5	2.5
Solids, Suspended Ignited	(E3188)	mg/L	-	0.5	2.5
Solids, Total	(E3188)	mg/L	-	2.0	10.0
Solids, Total Ignited	(E3188)	mg/L	-	2.0	10.0
Sulphate	(E3004)	µg/m ³ SO ₄	14.7	0.1	0.5
Sulphate	(E3013)	µg/g		0.5	2.5
Sulphate	(E3172)	mg/L SO ₄	100	0.5	2.5
Turbidity	(E3311)	FTU	2000	0.05	0.25



(7920)

TD/380/P47/2000/MOE

TD/380/P47/2000/MOE

Wright, Bernard

Performance report

General Chemistry ajgr

C.1 a aa